

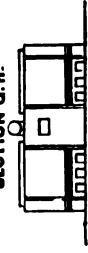
A
PRACTICAL TREATISE
ON THE
MANUFACTURE OF SULPHURIC ACID.

LONDON :
GILBEET AND RIVINGTON, PRINTERS,
ST. JOHN'S SQUARE.

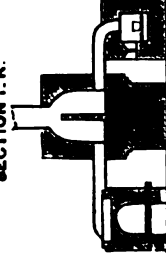
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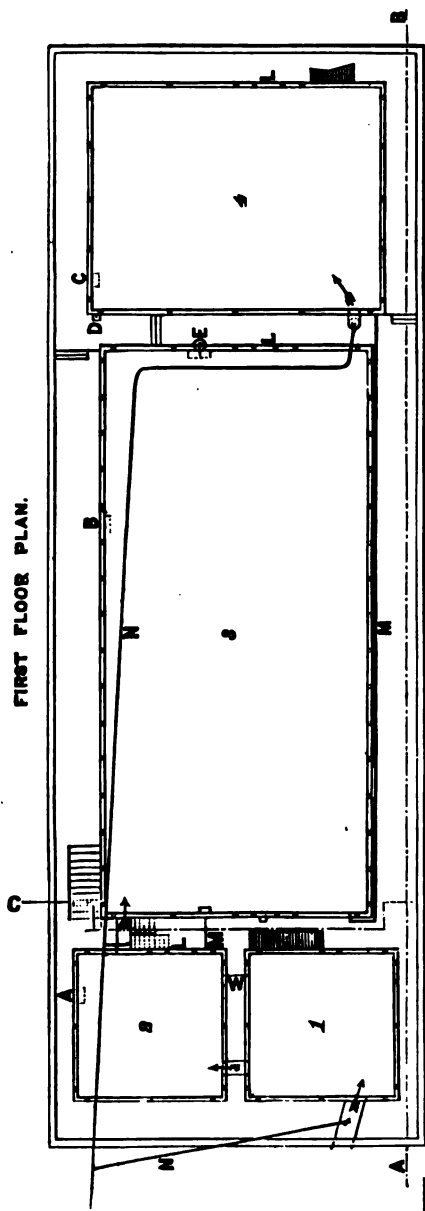
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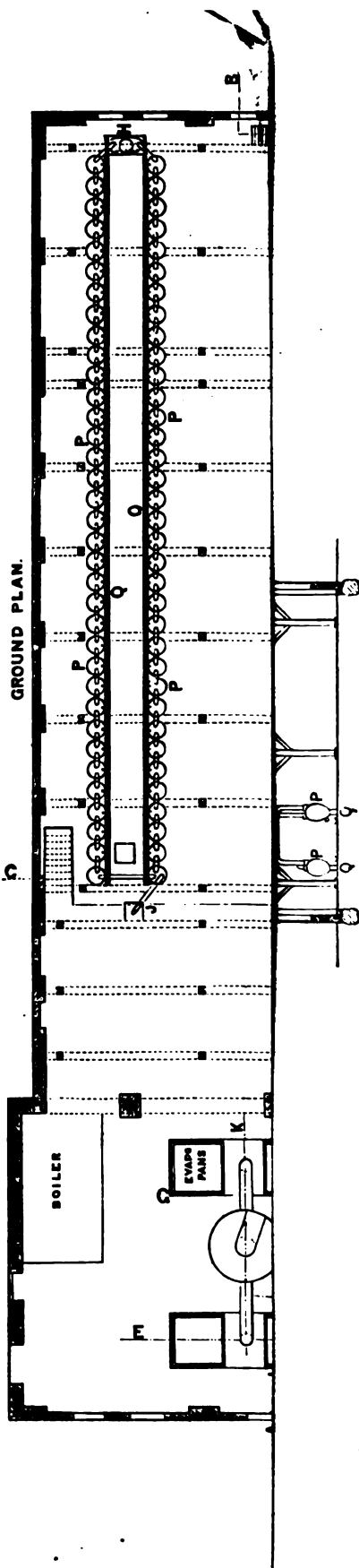
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FIRST FLOOR PLAN.



GROUND PLAN.



FRONTISPIECE.

A
PRACTICAL TREATISE
ON THE
MANUFACTURE OF
SULPHURIC ACID.

BY
ALFRED G. LOCK AND CHARLES G. LOCK,
CONSULTING CHEMICAL ENGINEERS.

*With 77 Construction Plates, drawn to Scale Measurements,
and other Illustrations.*

NEW YORK
PUBLISHED BY
L. B. S. S. S.
London:

SAMPSON LOW, MARSTON, SEARLE, AND RIVINGTON,
CROWN BUILDINGS, 188, FLEET STREET.

1879.

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A

PRACTICAL TREATISE

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INTRODUCTION.

THE present century has seen the birth and growth of more manufactures than, probably, the whole of the Christian era besides.

The production of sulphuric acid, first achieved about a thousand years since, has within very recent times waxed from a laboratory operation to gigantic proportions, and the chemical manufacturing trade of this country has attained an importance second to none but the coal and iron trades.

That sulphuric acid is the very life and soul of the chemical industry may be seen from a glance at the substances to whose manufacture it is essential, or at any-rate convenient. Principal among them is alkali, and though Hargreave's patent threatens to displace sulphuric acid by sulphurous oxide in this product, it will doubtless continue to hold its ground for some time to come. Then, again, artificial manures absorb an immense quantity of sulphuric acid, and this is a branch of industrial chemistry which must ever increase, for, even in the utilization of sewage, sulphuric acid must be used, if it be intended to save the most valuable ingredient—the ammonia. Also

for bleaching, dyeing, and printing; in telegraphy, for producing electric currents, and for electro-plating; for galvanizing, and wire-working; for cleaning iron, copper, and silver, for refining gold and silver, and separating gold from copper alloys. Further, in making alum; tin-plate and galvanized iron; in the manufacture of blacking, of sugar from starch, and of mineral waters. Again, for dissolving iron, and zinc, to procure hydrogen gas for balloons, &c., and for dissolving indigo, in the preparation of madder dyes, ether, collodion, nitro-benzol, nitro-glycerine, gun-cotton, wood-gunpowder and vegetable parchment, precipitating lime from molasses, extracting pure hydrocarbons from tars, removing grease from cotton and woollen goods; purifying oils; staining wood; and removing fungoids from casks, &c. Many acids, such as hydrochloric, nitric, phosphoric, boracic, fluoric, tartaric, citric, stearic, picric, &c., require sulphuric acid for their production. Likewise the many commercial sulphates, of ammonia, alumina, iron, copper, potassium, sodium (Glauber's salt), magnesium (Epsom salt), and mercury (for calomel, &c.), necessarily consume a large amount of sulphuric acid in their formation. In every chemist's laboratory it must have a place, and as Science advances, almost every day finds fresh uses for this most valuable, cheap, and powerful acid.

The fabrication of a product of such importance and demand seems worthy of a practical handbook. As manufacturers we felt the want of some such guide and as ex-manufacturers we now have the assurance to endeavour to supply the want by supplementing our own actual experience with every other information we can glean upon the subject.

We are especially indebted to Messrs. Johnson, Matthey, and Co., Hatton Garden, for the figures and details descriptive of their most recent improvements in platinum concentrating apparatus; to Messrs. R. Daglish and Co., St. Helen's

Foundry, for similar illustrations of their improved kiln-fronts and large nitre oven, and to Mr. William Glover, of Wallsend, for the sketch and particulars of the latest modifications effected in the Glover-tower.

To render the work as practical as possible and to bring the information down to the most recent date have been our great objects, and we trust that an indulgent public will be lenient upon the faults and shortcomings of which we have been guilty.

We desire to record our appreciation of the valuable columns of the *Chemical News*, where many interesting questions have found ventilation; and we cannot conclude these prefatory paragraphs without testifying to the liberality of our publishers, who have generously acceded to our views in allowing the work to be profusely illustrated, and have permitted us to add a number of plates and many pages of matter whilst the book was actually passing through the press.

CHAPTER I.

KILNS.

Rationale of Manufacture. — The commonly accepted *rationale* of the manufacture of sulphuric acid, is that the oxidation of sulphurous oxide (derived from the combustion of brimstone or pyrites) into sulphuric acid, takes place (in the chamber or other apparatus constructed for the purpose) under the influence of vapour of water (steam), and in the presence of atmospheric air, at the expense of part of the oxygen of nitric acid vapour; the nitric acid gas is thus converted into nitric oxide, but immediately appropriates a new portion of oxygen from the air introduced for that purpose, and assumes its former state, only to be again robbed of oxygen as before. The process is continuous, and should be regular, and in theory, one portion of nitric acid gas once supplied, should suffice for the production of sulphuric acid, *ad infinitum*, so long as the supply of the other necessary agents—sulphurous acid, air, and steam—is constantly renewed. But in *practice* it is not so, as a constant waste of nitric acid gas is caused by the irregularities necessarily incident to manufacture on a large scale.

Sources of the Gases.—Besides air and steam (which latter may be derived from a boiler or other generator) the two gases necessary to the production of sulphuric acid—sulphurous acid gas and nitric acid gas, require special contrivances for their proper formation, according to the sources whence they are derived.

To begin with the sulphurous acid. This may be obtained from native brimstone, containing from 90 to 98 per cent. of sulphur; from natural metallic sulphides, of which the most common are those of iron and copper, commonly known as "pyrites," and containing proportions of sulphur varying from 30 to nearly 50 per cent., and from other materials.

Brimstone was used in the manufacture on a grand scale long before pyrites were thought of, and there is no doubt that, except in the matter of price, it possesses many advantages over the now more commonly employed pyrites; for example, it makes a much purer acid, causes far less wear and tear of plant, leaves scarcely any waste residue after burning, requires less nitre for the conversion of its sulphurous acid into sulphuric acid, and the kilns for its combustion are more easily and cheaply erected. But pyrites are in such abundance and so much cheaper than brimstone that, despite their disadvantages, they have come to be very generally adopted, in cases where the sulphuric acid is required for manure or alkali manufacture, and when the presence of arsenious acid (a constituent of all pyrites) is not an insurmountable evil.

Removing Arsenic.—It will be found in practice that sulphuric acid, which is intended for galvanizing, bleaching, dyeing and printing purposes, and for the manufacture of mineral waters and sulphate of ammonia, as well as in any other case where purity is necessary, must be made from brimstone. It is true that it is claimed for some pyrites that they contain no arsenic, but the following figures, which are published by Mr. H. A. Smith, as the result of numerous researches, prove that such statements cannot be depended upon.

In these tables No. 1 shows the percentages put forward by the pyrites merchants, while No. 2 gives the results actually obtained by assay.

Name of Pyrites.	Arsenic per cent. Mean No. 1.	Arsenic per cent. Mean No. 2.
Spanish	0·21 to 0·31	{ Tharsis 1·651 Mason's 1·745
Belgian	trace	·943
Westphalian . . .	trace	1·878
Norwegian	none	{ hard 1·649 soft 1·708
Irish	0·33	—
Cornish	0·32	—
Italian	trace	—
Swedish	trace	—
Cleveland	—	—

He further says,—“In some Irish ores that have come under my notice, the result has been very different from that given in the table. I have analyzed ores containing 2 per cent., and even 2·3 per cent. arsenic, and one of our most successful acid manufacturers assures me that he has often had Irish ores containing even a larger percentage than that which I have given.”

Below we give a short *résumé* of the newest methods attempted by him for getting rid of this ever-troublesome arsenic.

The first plan consisted of introducing sulphuretted hydrogen, in a gaseous state, into the acid by means of a pipe connecting with a pan 24 ft. by 3 ft., by 3 ft., and running throughout its whole length, perforated with fine holes through which the gas bubbled into the acid. When the precipitation was complete, the acid was filtered through coke, which withheld the sulphide of arsenic. The faults of this method are its expensiveness and the nuisance created by the sulphuretted hydrogen.

A modification of the above was the introduction of finely ground sulphide of iron into the acid. As a purifier it acted well, in fact admirably, and was all that was needed

for acid intended for use in galvanizing and similar work, but the acid could not be used in dyeing, bleaching, printing, &c.

The substance next tried was sodium sulphide, added to a known quantity of sulphuric acid, in excess of the previously determined arsenic tri-oxide. Coke was again used to filter off the precipitated matters. "The results obtained by this method were very satisfactory, and the expense was extremely moderate, 100 gallons of sulphuric acid giving only a very minute trace of arsenic after being subjected to this process. . . . The only difficulty lies in the method to be employed and the means of getting rid of the escaping sulphuretted hydrogen which has been allowed to be present in excess."

Another plan tried was the addition of common salt to the retorts during concentration, but this had no practical result, and we trust Mr. Smith will not repeat that experiment till he has lived to make many more researches into the chemistry of sulphuric acid making.

Passing hydrochloric acid gas though the acid was found too expensive. So far "the use of sodium sulphide answers the purpose to a more perfect degree than any other process."

Another plan which has come under our notice is as follows. The chamber acid at 106° Tw. was heated in a leaden pan to 70°—80° (? Centigrade) with the necessary amount of hyposulphite of sodium. If well stirred, the whole of the arsenic will float on the top of the acid in the form of thin flakes of sulphide of arsenic. In this experiment the arsenic existed in the acid to be treated in the proportion of .098 to .004 per cent., and the purified acid was found to contain .3 to .4 per cent. of sulphate of soda.

By a modification of the above process, Wagner avoids the drawback of the formation of sulphate of soda in the de-arsenized acid. Using a mixture of hyposulphite of sodium and chloride of barium, he gets a deposit of sulphide of arsenic and sulphate of barium at the bottom of the

vessel. It would, however, be almost impossible to carry out this method in an ordinary leaden concentrating pan, as the deposit would have to be removed from the bottom of the pan on each occasion, which would be a most troublesome operation.

Selection of Pyrites.—In the selection of sulphur ores, it is well to reject any which contain a quantity of *earthy carbonates*, recognizable by their effervescence under dilute acids, for such ores would give off carbonic acid in burning, and this gas would altogether vitiate the chemical process that ought to go on in the chambers. Also there should be taken into consideration the character of the ore, whether compact or crumbling, as on that will greatly depend the proportion of dust created in breaking.

Preparing the Pyrites.—Before burning the pyrites they must be broken into pieces not exceeding $1\frac{1}{2}$ cubic inches in size. This may be done either by a stone-breaker worked by steam, where very large quantities are used and labour is dear, or by hand when the consumption is not so great.

The class of stone-breakers most generally adopted in this country is Blake's, costing about 180*l.* for the size 15 in. by 7 in. between the jaws. Messrs. Sievers and Co., of Kalk, near Deutz, Prussia, make a stone-breaker which is in general favour among Continental manufacturers. It has the advantage of being very much cheaper than Blake's. E.g. :—

Size between the jaws.	To break tons per day.	Price at Cologne.
$7\frac{1}{2}$ in. $\times$ 4 in.	10	£25
9 in. $\times$ 6 in.	20	33
12 in. $\times$ 9 in.	30	42

The makers say that the largest size named takes lumps 12 in. by 9 in., and reduces them to from $\frac{1}{2}$ in. to 1 in. cube, making about 10 per cent. of dust. It can be regulated to break into larger pieces if required, when the amount of dust would be less. Weight of this size machine about 4500 lbs., requires 5 h.p.



FIG. 1.

Some firms employ boys for the purpose, using cast steel hammers manufactured by H. Rossell and Co., Sheffield, of the shape shown in fig. 1. A sledge hammer 13 lbs. in weight (1s. per lb.) is first used to reduce the large pieces to a manageable size for the smaller hammers of $3\frac{1}{2}$ lbs. weight (1s. 3d. per lb.). Hand-breaking produces much less dust.

Product, Theoretical and Practical.—Theory requires that 306 parts of sulphuric acid at 66° Beaumé (say 1.845 sp. gr., or 169° by Twaddell's hydrometer) should result from the combustion of 100 parts of sulphur burnt, whether contained in brimstone or pyrites, but in practice this standard is never reached. One of the best authorities on the subject says, that the figures vary from 275 (lowest) to 300 (highest), giving an average of about 292. A very large manufacturer using brimstone and with very perfect appliances has never exceeded 300, and his average is about 296. A Glasgow maker, with very large chambers reaches nearly to 300. Any one using pyrites and having small chambers may be well satisfied if he obtains 290, and anything above that shows extremely good work.

Proportion of Nitre.—When pyrites are used more nitre is required than in the case of brimstone. Eight pounds of nitre for every 100 lbs. of brimstone is an ample allowance, while 8 lbs. of nitre for every 100 lbs. of sulphur contained in the pyrites (if found sufficient) is not at all exorbitantly high, even reckoning that it is in reality 8 lbs. to every 93 lbs. or 94 lbs. of sulphur extracted from the pyrites, seeing that from 2 to $7\frac{1}{2}$ per cent. is left in unburnt. In fact some manufacturers require 9 lbs. and even 10 lbs. of nitre for every 100 lbs. of sulphur burnt out of the pyrites, that is to say where no Gay Lussac or Glover's tower, or other apparatus is in use for the recovery of the escaping nitrogen compounds. Where these are employed, the consumption of nitrate soda may

vary from 1 to 5 per cent of the sulphur converted into sulphuric acid.

Brimstone or Pyrites.—To Messrs. Richardson and Watts we are indebted for the following details of the cost of materials and labour to make the same quantity of acid from sulphur and pyrites, which possess some value as the result of the practical working of chambers by a manufacturer of great experience. The prices of the materials are assumed to show the relative value of the ores as compared with sulphur.

SULPHUR.					£	s.	d.
t.	c.	q.	lbs.				
5	10	0	0	Sulphur @ £7	38	10	0
		5	2	Nitrate Soda @ 18s. . . .	4	19	0
				2 men @ 21s. per week . .	2	2	0
					45	11	0
BELGIAN PYRITES.							
13	15	0	0	Pyrites @ 45s.	30	18	9
		6	2	Nitrate Soda @ 18s. . . .	5	18	9
				8 men @ 21s. per week . .	8	8	0
					45	5	6
IRISH PYRITES.							
18	0	0	0	Pyrites @ 33s.	29	14	0
		6	2	Nitrate Soda @ 18s. . . .	5	18	9
				9 men @ 21s. per week . .	9	9	0
					45	1	9

Methods of Estimating Sulphur.—Many disparities have occurred in analyses of sulphur ores through the use of a Wedgewood mortar for their reduction to powder. A steel mortar may first be employed in the operation, and then they should finally be rendered impalpable in an agate mortar.

For estimating the proportion of sulphur contained in a metallic sulphide, Pelouze mixes 1 gramme of the finely powdered sulphuret with 5 grammes of calcined carbonate of soda, 2 grammes of chlorate of potash and 5 grammes of common salt, and exposes the mixture to a dull red heat for about 10 minutes in an iron ladle. When

the mass has cooled sufficiently it is carefully exhausted with warm water, and the undecomposed carbonate of soda determined volumetrically by means of sulphuric acid in the usual manner. The difference between the amount of sulphuric acid necessary to decompose the 5 grammes of carbonate of soda and that actually used, gives by calculation the quantity of sulphur in the ore.

A simpler plan is to oxidise with concentrated nitric acid, or with nitric acid and chlorate of potash, using chloride of barium to precipitate the sulphuric acid, and estimating.

According to Anthon the specific gravity is a safe guide as to the quality of the pyrites.

Nitrogen Compounds Employed.—The oxides of nitrogen necessary for assisting in the conversion of the sulphurous gases into sulphuric acid may be derived either from nitric acid or from the nitrates of soda or potash. Continental manufacturers prefer the former plan, and in France and Germany it is almost universal, while the latter method is equally common in this country.

Nitrate of potash contains a smaller proportional weight of nitric acid than nitrate of soda does, and the latter (commonly, though erroneously, called “nitre”) is also so much the cheaper of the two that it is, practically, the only salt employed as a source of nitric acid in this manufacture.

Commercial “nitre” is not quite pure, but the amount of its impurities—principally common salt—should never exceed 5 per cent., and nitrate containing a large amount of foreign matters should be rejected, especially in those cases where platinum vessels are used for concentration, as that metal is liable to suffer corrosion by the presence of large quantities of sodium chloride.

Brimstone-kilns.—For burning brimstone the only erection necessary is a room or oven of brickwork with an arched roof. The size of the kiln will be regulated by that of the iron plate or flooring, and this latter by the quantity

of brimstone it is proposed to burn every 24 hours. It may be a rectangular iron tray measuring 8 feet by 4 feet, and surrounded by a vertical edge or border standing about 1 inch high. This border should be at right angles to the plate on three sides, but on the fourth side, the one nearest to the door, it should incline outwards at an angle of about 45° , for the more convenient raking out of the residual dirt and ashes left after the combustion of the brimstone. The door need be simply a cast-iron plate, sliding in grooves in an iron framework, and counterbalanced by a weight hanging at the end of a chain passing over pulleys.

The iron plate does not cover the whole of the floor, but the brimstone should be allowed to burn on it alone. The object of making the kiln rather larger than the plate is to avoid, as much as possible, all risk of sublimation. To this end also there is a constant current of air passing beneath the plate and up a small chimney beside the kiln. Four kilns with plates of the size mentioned above will easily convert into sulphurous acid 1 ton of brimstone per 24 hours. The quantity for each kiln of the series is divided into 6 equal portions, one of which is added every 4 hours.

The starting of the kiln is effected by lighting an ordinary fire under the iron plate, and continuing it until the plate becomes sufficiently hot to assist the ignition of the brimstone when placed on it.

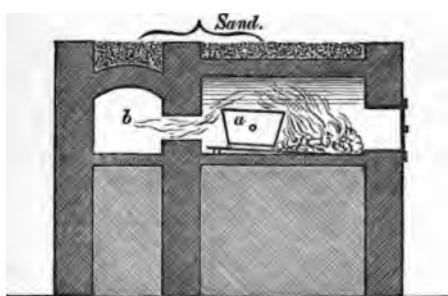


FIG. 2.

Another Form.—The following plate (fig. 2) and description refer to a form of brimstone-kiln which we found to answer admirably. The floor is formed of fire-tiles resting upon a compact bed of rubbish filled in between the

retaining walls, no iron plate being necessary. The door is of the ordinary form, hung on hinges. The nitre-pot, *a*,

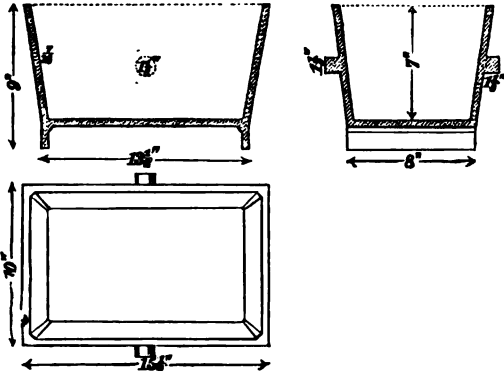


FIG. 3.

is placed towards the back of the kiln on a sloping iron stand, which supports it an inch or so off the bed. The gases arising from the combustion of the brimstone and from the contents of the

nitre-pot are conducted by means of a flue, *b*, into a brick stalk connecting with the chambers. The top is covered with a layer of sand to conserve the heat. A kiln of this sort, measuring 3 feet 6 inches square, and 1 foot 6 inches or 1 foot 9 inches high, will properly burn about 26 lbs. of ordinary "best thirds" brimstone in every three hours. No chimney nor fireplace is required for starting it. This is effected by introducing 60lbs. or 70lbs. of brimstone and mixing with it a quantity of glowing cinders sufficient to ignite it, the door being left partially open in order to create a draught. A larger quantity than the regular charge must be kept burning in this way, until the kiln is hot enough to burn off the proper charge in its allotted time, with the door shut and without any artificial aid. A handful or two of nitre sprinkled over the first two or three charges of brimstone assists the combustion and improves the gas. A measured quantity of nitre with a proportionate quantity of acid, which varies according to circumstances, as will be described hereafter, is placed in a cast-iron pot (fig. 3), which is lifted in and out of the kiln by the handle (fig. 4).

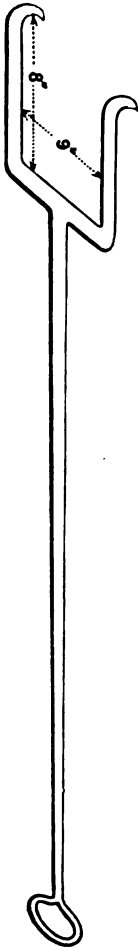


FIG. 4.

Until the kiln has attained its proper heat the nitre will not yield all its nitrous gas, therefore at first the nitre-pot is made moderately hot before being charged.

The difficulties attending the ordinary methods of burning brimstone in the manufacture of sulphuric acid, either in brick or in cast-iron furnaces, consist chiefly in obtaining the proper admission of atmospheric air, to provide for the combustion of the sulphur, and its subsequent conversion into sulphuric acid, without, on the one hand, producing so high a temperature as to cause sublimation, and, on the other, not having the heat requisite to burn the whole of the sulphur from its impurities; whilst in both instances, in consequence of the frequent interruptions in the operation caused by the charges being necessarily small and quickly burned off, a larger quantity of atmospheric air is admitted into the chambers than is requisite, and by diluting the gases, retards the formation of sulphuric acid.

Blair's Kiln.—To remedy these defects, a furnace has been planned and erected by Harrison Blair, of Kearsley, in Lancashire, in which the sulphur is partly burned, and partly sublimed in one compartment, and the combustion completed in another, by the addition of a further supply of atmospheric air. By this arrangement, although both compartments are at a red heat when the furnace is in full action, sublimation is next to impossible, and at the same time the operation of burning is so nearly continuous, that it is suspended only once in 24 hours, for the purpose of withdrawing the residuc.

In the accompanying fig. 5, *A* is the bed of the brimstone furnace, which is slightly dished and sloping to within 2 feet of the door, where there is a raised floor for the purpose of permitting the residue, which is drawn from the floor, to remain until the sulphur is completely burned out, when it is pulled out and replaced by that which, at the same time, is drawn from the floor, this being done once every 24 hours. The door, *B*, is a loose iron plate placed in

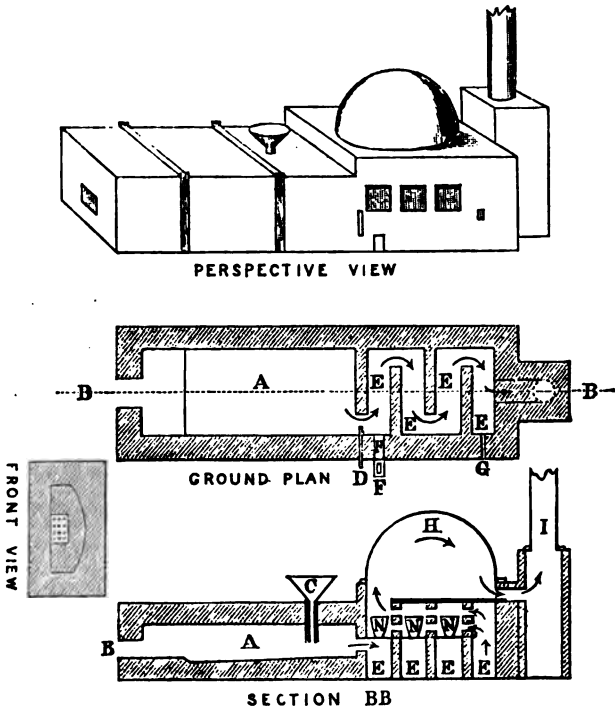


FIG. 5.

an iron frame, a little out of the vertical, so as to make an almost air-tight arrangement, and easily removable when required. This door is perforated with a number of holes, which can be closed by a slide, so as to regulate the admission of air into the furnace. The brimstone is fed through a hopper, *C*, from which an iron pipe, 7 inches in diameter, is continued to within 6 inches of the floor of the furnace, this pipe being protected by one of larger diameter.

A fire-clay damper, *D*, regulates the passage from the brimstone furnace to the combustion oven by opening or closing which, a larger or smaller quantity of brimstone may be burned in a given time.

The sulphurous acid mixed with the vapour of sulphur, passing from the brimstone furnace to the combustion oven, *E*, is met by a current of atmospheric air admitted by

the aperture, *F*, which is furnished with a damper of 3 inches by 8, to regulate the quantity required for perfect combustion of the vapours of sulphur; this is ascertained by taking out the small stopper at *G*, when no flame should appear by the admission of air through the aperture. The roof of the combustion oven is of tiles, which also form the floor of the nitre oven, supported on dwarf walls between which the gases traverse, in the direction of the arrows, into the nitre oven, where reticulated brick walls support the tiles forming its roof, and permit the passage of the heated gases over the nitre dishes, *N*. These dishes are renewed every 2 hours in alternate sets, iron doors corresponding with each department providing for their removal, so that a set remains 6 hours in the oven.

The gases now mixed with those from the decomposition of the nitre, pass under a cast-iron dome *H*, for the purpose of being deprived of a portion of their heat, and thence by a cast-iron chimney, *I*, 24 feet in height, to a small cooling chamber 6 feet wide, 18 inches high and 18 feet long, the roof and floor of which are covered with water, which communicates with the sulphuric acid chamber.

Steam has recently been admitted along with the air into the combustion oven, and is supposed to cause a more rapid formation of sulphuric acid.

In a furnace of the dimensions 2 feet by 4½ feet, 26 tons of brimstone have been satisfactorily burned in a week, and the same furnace, by admitting less atmospheric air, will burn only 5 or 6 tons in the same space of time; it has also been found that a much larger quantity of brimstone may be safely burned in the same space of chambers, than before this plan of furnace was adopted.

Respecting this kiln, Mr. Blair's successors write us (August 5th, 1878), "We have made no further alterations in it, but may say that we are still working the furnace, which works very satisfactorily."

Its advantages and drawbacks.—We know manufacturers who use these kilns, and they speak favourably of them so far as regards the economy of space, materials, and labour in building, but it is objected that a certain amount of sublimation of the sulphur does take place, and it is questionable whether more than half the maximum quantity stated above could be properly burnt in a furnace of the dimensions indicated in seven days.

Pyrites Kilns.—It must not be supposed that all classes of Pyrites can be treated effectually and economically in the same kind of kiln. A pyrites poor in sulphur requires to be roasted in a great mass, in order that sufficient heat may be kept up in the kiln to ignite each charge as it is introduced, while a rich ore, on the other hand, if burnt in such a kiln, would in all likelihood fuse into a solid slag and cause endless trouble and expense, and we have even known cases where the kiln had to be pulled down in order to get out the clinker.

For Norwegian Pyrites.—The following set of kilns (fig. 6) were used by us for many years for Norwegian iron pyrites containing 44—46 per cent. of sulphur, and gave complete satisfaction.

All kilns for burning pyrites must be built throughout of fire-bricks laid in fire-clay, which latter has been well mixed up in tubs into a thin fine batter, twelve hours or more before use. The joints must be as close as possible, seven bricks laid should rise only eighteen inches.

A B C D the four kilns in plan, elevation and section.

C' a transverse section of one.

E nitre-pot oven, being part of the flue of the kilns leading to chambers.

a cast-iron bearing bars 4 inches $\times$ 1 $\frac{1}{4}$ inches.

b wrought-iron fire bars 1 $\frac{1}{2}$ inch square, on which are turned three journals where they rest on the bearing bars.

c wrought-iron bars 1 inch square, to support brick-work, &c., of the front of the kiln.

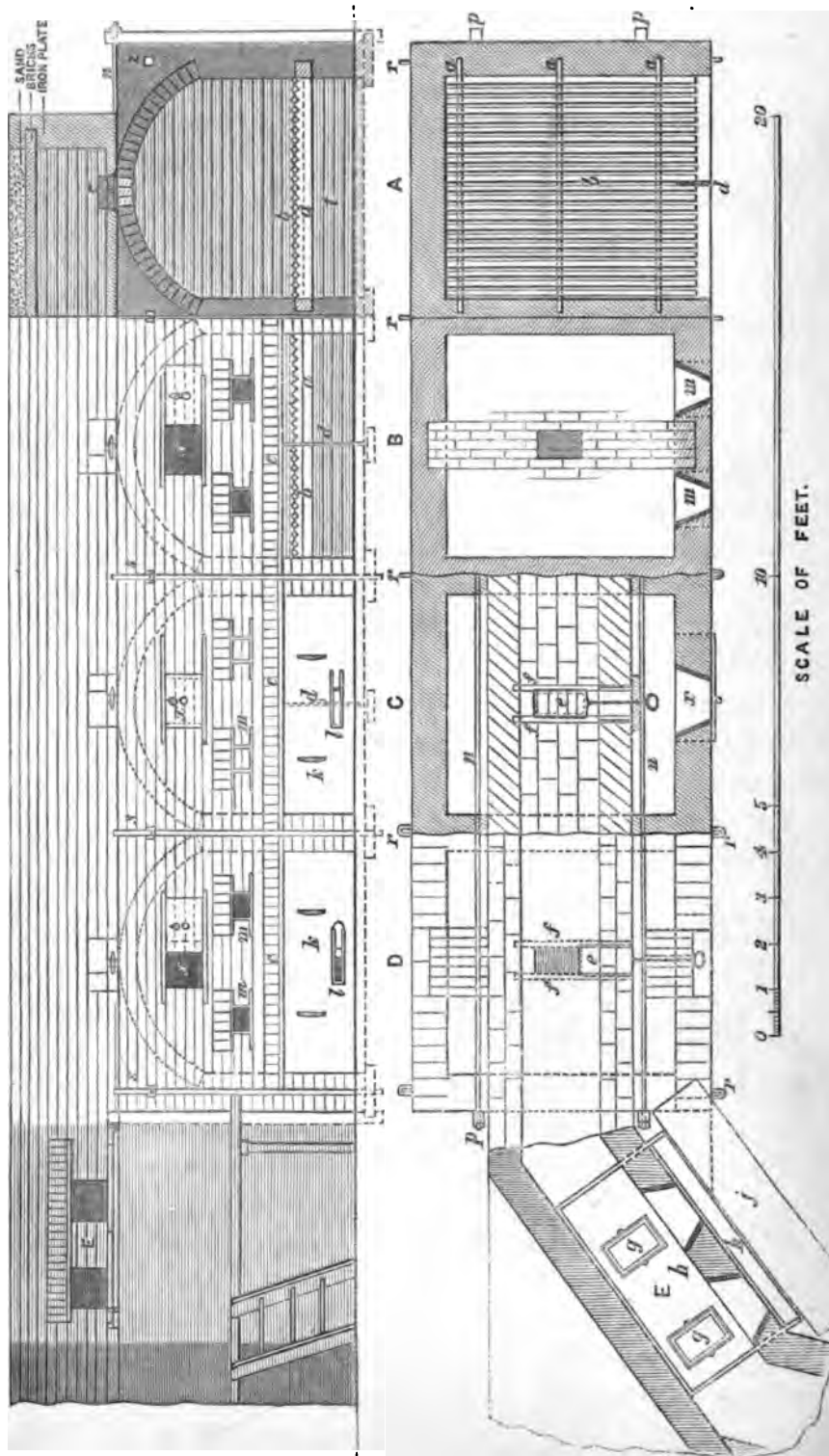


FIG. 6.

d cast-iron standards for centre support to *c*.

e dampers to regulate draught from kilns to flue, formed of fire-bricks, clamped with an iron band, sliding on the irons *f, f*.

g nitre-pots.

h iron plate with back and side edges turned up about 1 inch, forming the bed of the nitre-pot oven.

j iron table on which to rest nitre-pots during process of charging.

k sheet-iron damper closing lower front of kilns, being removable at pleasure.

l ventilators in *k*.

m clinkering holes.

n wrought-iron tie bars 1 inch square, running the whole length of the kilns at top and bottom on each side, and furnished with bows into which are wedged upright cast-iron bars 3 inches $\times$ 1 inch, *p*.

r transverse tie bars, 5 at the top and 5 at the bottom, 1 inch square, furnished with bows and uprights, *s*, as the longitudinal ones.

x charging doors.

z hole left in brickwork to receive iron tie bars.

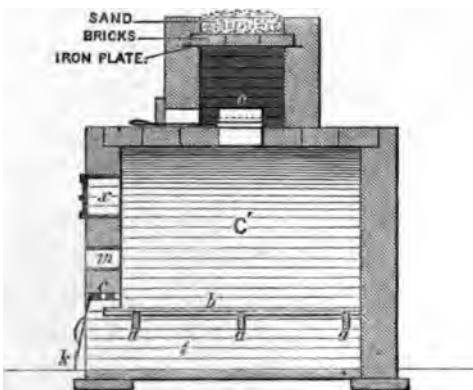


FIG. 6. Section.

Starting and Working the Kilns.—Previous to lighting up the kilns, their communication with the chambers must be temporarily suspended and a way opened into the air for the free escape of the smoke. The dampers,

e, are drawn out, the doors, *k*, removed; the doors of the nitre-pot oven, *E*, shut; and the doors, *m*, also shut. Through the doors, *x*, the kilns must be filled nearly up to

the level of the doors, *x*, with coarse rubble (preferably brick-bats) which, while sufficiently hard to remain in a whole state and allow of an abundant current of air to pass upwards between them, so long as that is necessary, will yet easily crush by the revolution of the bars, *b*. On the top of this is spread a layer of glowing cinders, covered in turn by 2 or 3 inches of coke which has been broken into pieces about 2 inches square. The doors, *x*, are then closed and the fire left to burn with the occasional addition of more coke as required, until the whole of the interior of the kilns is thoroughly hot, generally about 12—18 hours. When a sufficiently high temperature is reached the exit to the open air is closed and the connexion with the chambers restored, the doors, *k*, replaced, and a quantity of the previously broken pyrites is introduced through the doors *x*, and levelled with the iron rake, the door is then

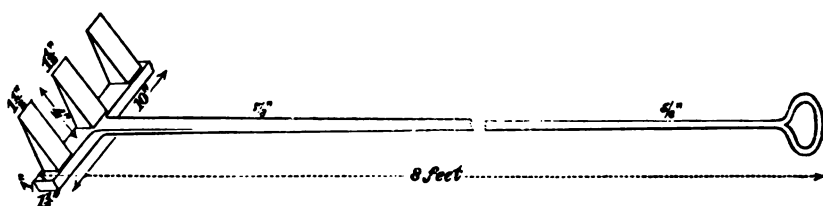


FIG. 7.

shut and luted with clay, and a nitre-pot duly charged, is inserted in *E*. All the doors, *x*, *m*, *k*, as well as the doors of *E* are luted with clay when not open.

Accurate details of the quantities of pyrites and nitre to be used during the first 24 hours cannot be given, as they will depend entirely upon the behaviour of the kilns, the principal object being to bring the kilns into the proper condition for burning off the regular charge in the appointed space of time. We will only remark that it will be found advisable to use as much as twice the usual quantity of nitre during the first day (whether in the case of brimstone or pyrites kilns, of whatever form they may be), gradually

reducing it as the appearance of the gas in the chambers may dictate.

Charge required, and how Proportioned.—The size of the kilns is always calculated after the quantity of material to be burned in a given time and this bears a constant relation to the cubic capacity of the chambers. Whatever the quantity, it must be regularly and unvaryingly supplied. The proper charges for these kilns, which were constructed to supply the set of chambers hereinafter to be described in detail (fig 17). are as follows :—viz., 2 tons of Norwegian iron pyrites, containing 45 per cent. of sulphur in every 24 hours. Each kiln is charged in rotation with $\frac{1}{8}$ of the total quantity to be burned in 12 hours, i. e. $2\frac{1}{2}$ cwt., at intervals of $1\frac{1}{2}$ hours. Thus kiln A is charged, say, at 7 a.m.; B. at 8.30; C, at 10; D. at 11.30; A is again charged at 1 p.m., &c.; so that each kiln has 6 hours in which to burn its charge. The ore in the kiln must never be allowed to rise above the level of the bottom of the door, *x*. This level is maintained by means of the revolving bars, *b*. Just before a charge is put into the kiln

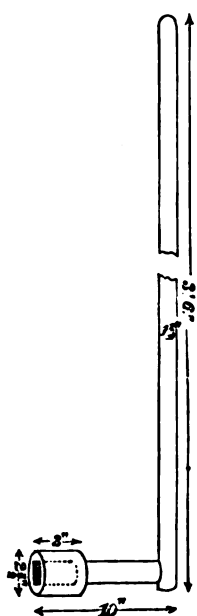


FIG. 8.

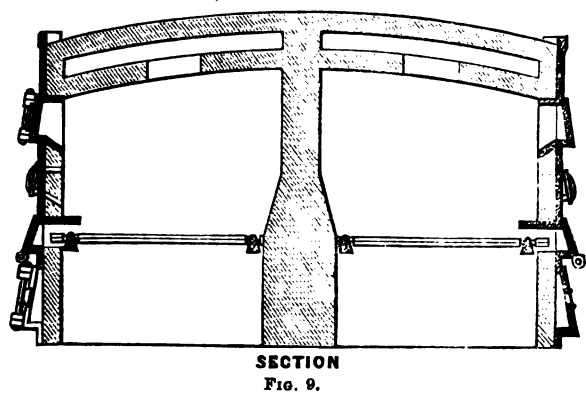
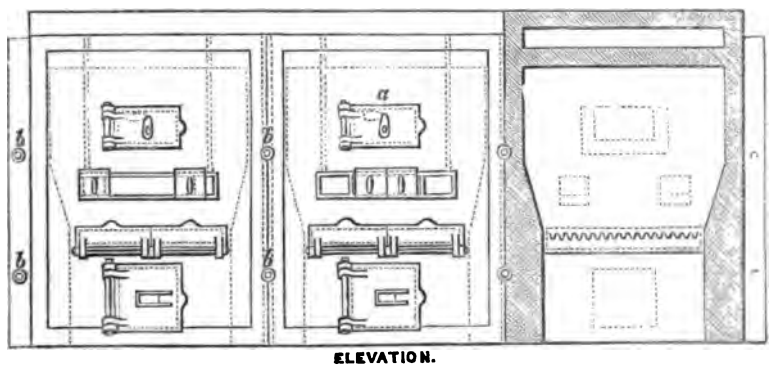
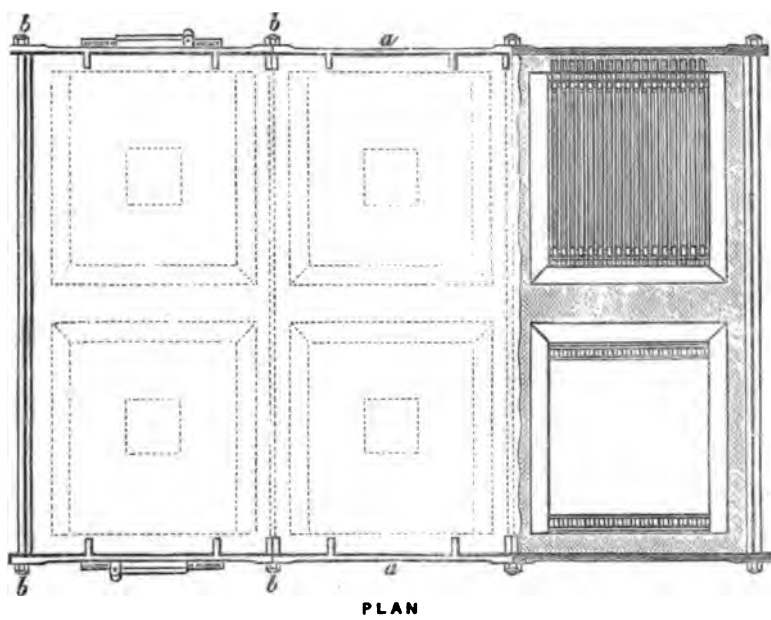
a handle is affixed to the end of each bar in succession, and by it they are turned backwards and forwards until so much of the burnt ore has thus been made to fall through that sufficient space is left for the new charge. The burnt ore is drawn out by means of a long-handled shovel at *t*, and carried away to the refuse heap in an iron wheel-barrow. It is often necessary to insert a long crowbar at *m*, in order to break up any clinker that may have formed and which would not otherwise pass out at the bottom.

By means of the dampers, *e*, any kiln of the series can be stopped if necessary. Too great a temperature in the kilns

can be checked by the ventilators, *l*, while too low a temperature is corrected by a little broken coke being mixed with the charge, as well as opening the dampers. One nitre-pot containing about $8\frac{1}{2}$ lbs. of nitre and an equal weight of sulphuric acid of the strength 125° to 130° , by Twaddell's hydrometer, is put into the oven, *E*, just before each kiln is charged. Three of these pots are kept in use, two being always in the oven, and a burnt-off one being drawn out at each charge to make room for the fresh one. The above proportions of nitre and acid form a good cake, that is to say, all the nitrous gas will be extracted, leaving behind a soft white mass of sulphate of soda which becomes hard on cooling, and is removed from the pots by means of a broad chisel bar. When the nitrous gas has not been properly eliminated the cake gives off heavy red fumes on being turned out. This happens when the sulphuric acid has been deficient in quantity or strength.

Daglish's Fronts.—Messrs. Robert Daglish and Co., St. Helen's Foundry, Lancashire, make iron fronts for pyrites kilns which are constructed as shown in the following plan elevation, and section (fig. 9). The fronts, *a*, are tied together by the bolts, *b*, passing through the brickwork. Every care is taken that the joints of the doors are made airtight by means of planed faces, and thus the trouble of clay luting is avoided. Any number of kilns may be built together. They are immensely strengthened by the iron-work and its addition is to be highly recommended, especially when the pyrites in use has a tendency to slag, causing great strain and wear and tear on the brickwork when the clinker is broken up.

For poor Pyrites as Irish.—The following kiln (fig. 10) is well adapted for the roasting of poor non-cupreous ores, free from bituminous matter, such as Irish pyrites containing about 30—35 per cent. of sulphur, as they require to lie in a deep mass in order to retain sufficient heat for their combustion.



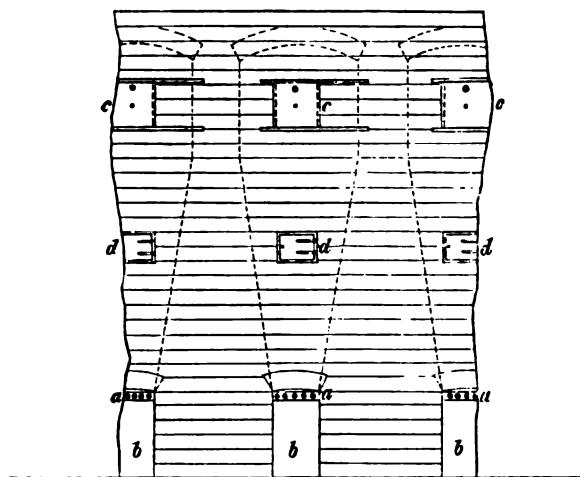


FIG. 10.

The body of broken pyrites rests on square iron bars, *a*, which are made to revolve as shown in figure 6, while the air rushes up from the ash-pit, *b*, and wends its way through the crevices and interstices of the mass to feed the combustion. The kilns are built in sets arranged back to back, and having a gas passage between them common to both, by which the sulphurous gases are conveyed over the surface of nitre-pots at the end of the series, and the mixed gases are then conducted to the chambers. Fresh supplies of mineral are introduced at regular intervals through the charging door, *c*, which is kept closed except for this purpose. Another orifice exists at *d*, which is for the purpose of inserting an iron bar for stirring up the burning mass. Sometimes the combustion becomes so rapid and the heat so great that the pyrites becomes fritted or enters into a semi-fused state, occasionally even the whole body may become fused together by its points of contact into one solid slag. It is then that the orifice, *d*, becomes necessary for admitting a crowbar with which to separate the mass and create a passage for the draught. The orifice is furnished with an iron door which is made to fold back flush with the face of the kiln when open, when not in use it is kept shut. The charging door, *c*, slides in grooves in an iron frame and is provided with a button

cast on it with which the bar may come in contact when pushing it open or shut. There is also a small hole pierced in the door and covered with a riveted movable strip of iron. By moving this little curtain aside it is possible to watch the progress of the combustion in the kiln without letting in a current of superfluous air.

For Coalbrasses.—Many coal-fields yield a quantity of bituminous iron pyrites which in England are locally called “Coalbrasses,” and being considered almost as a waste product, may often be had for a very low price. They contain a varying proportion of sulphur up to 36 per cent. This is the highest standard we have met with, and the average percentage would be considerably lower. They are objectionable only on account of giving the acid a black colour, a matter of no consequence in the great majority of uses to which the acid is applied.

The following description and figure (11) refer to the kilns which we erected for burning this material.

1 plan of the kilns.

2 3 side and end elevations.

4 5 longitudinal and transverse sections.

A the bed, is formed of wrought-iron bars, 2 in. wide by $\frac{1}{2}$ in. thick, laid side by side and kept at a distance of $\frac{1}{4}$ in., one from another by projections hammered out on the edges $\frac{1}{4}$ in. wide, the projections meeting on

B cast-iron pipes, nine in number, $\frac{3}{4}$ in. thick, with an interior diameter of 3 in., open at both ends to admit air, supporting the bed, *A*, built into the outside walls and sustained in the centre by

C cast-iron bar 3 in. square, resting on

D brick arches.

E longitudinal tie-bars, four in number, passing through the whole length of the kiln at top and bottom, and having the upright cast-iron bars 3 in. by 1 in., *L*, wedged into bows turned on their ends, and catching the flanges of the door-frame, *F*.

G upright cast-iron bars 3 in. by 1 in., supporting the

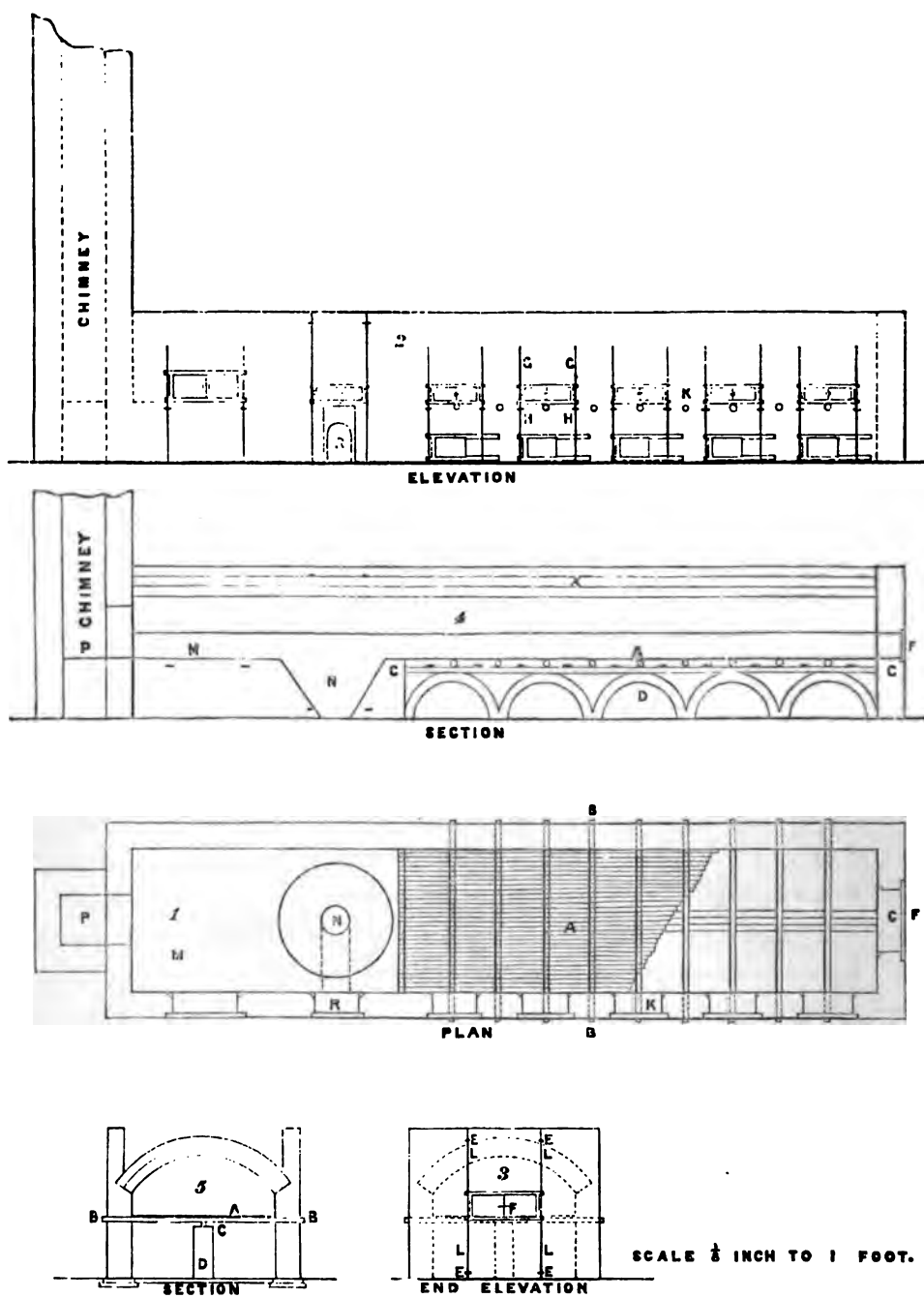


FIG. 11.

side walls against the outward pressure of the arch, *X*, catching the flanges of the frames of the side doors, *K*, and wedged midway into bows on the transverse tie bars, *H*.

M where the nitre-pots stand.

N pit where the kiln is discharged.

P flue for gases, leading to chambers.

This kiln is charged at the door, *F*, with a measured quantity of pyrites at regular intervals, care being taken that the ore shall cover evenly that portion of the floor, *A*, lying between the door, *F*, and the farther side of the first of the series of side doors, *K*. Before the next charge is put in, the first must be moved along from the place which it has occupied just within the kiln, to the farther side of the second of the series of side doors, *K*. This is effected by means of a paddle, a long iron shaft, having at one end a flat blade about 3 ft. long and 3 in. wide, which is inserted at each of the side doors, *K*, in rotation, always beginning with the charge lying farthest from the charging door. In due course the first charge will arrive at the inner end of the floor *A*, and will then be turned over into the pit, *N*, from which it can be drawn out at *R*, as it should by this time be entirely burnt off. A good rough test as to whether the kiln is doing its work may be got by breaking some of the lumps when drawn out. Kernels of raw ore found in the centre show that great waste is taking place. The bituminous matter which is intimately associated with this class of ore causes it to burn very freely, and therefore only a shallow kiln, such as the foregoing, is suited for it.

Pyrites Dust.—The breaking of the pyrites necessarily produces a certain amount of dust, varying according to the nature of the ore and the means employed for breaking it. This is, in some cases, a source of trouble by choking the draught in the kilns. When the dust will cake well it may be moistened, and made into balls to be dried steadily on the top of the kilns, or other convenient warm place, and then thrown into the kiln in that state along with the lump

ore. Another method of cementing the dust into balls, consists in mixing up the dust with a quantity of refuse oxide of iron from gas-works, which contains in itself a large proportion of sulphur. The use of clay to render the dust cohesive is to be condemned as importing a source of still more dust as soon as the heat has driven off the moisture.

French Kiln for Dust Pyrites.—In some parts of France and Belgium dust pyrites alone, or nearly so, is used for the manufacture of sulphuric acid. This requires a special kiln for its treatment, and that invented by Messrs. Olivier and Perret is in most general favour in those countries.

The ore dust is spread upon 6 trays through openings in the wall of the furnace which are kept closed during the process of the roasting. It is heated by the current of hot air and sulphurous acid passing up from the grate of the furnace where some lump pyrites is burnt. To check the evil which might arise from the over-heating of the furnace or from the absorption of the oxygen by the mass of lump ore roasting below, an aperture is made at about the centre of the tier of trays by which air can be admitted from the outside. As the calcining progresses the exhausted material is pushed along the trays till it falls at last into a vertical flue, and thence into the ash-pit, whence it is removed at a lateral aperture, as in the case of any other kiln.

The most general form of kiln used in France for ordinary pyrites is said by Dr. Quesneville to be a revolving kiln having a depth of about 5 ft. 11 in. (1.8 m.) and a surface of about 4 ft. (1.2 m.) in which the pyrites is burnt to within 2—3 per cent. of the sulphur contents. He considers that, as a rule, the kilns are not well proportioned to the chambers, and approves of the following dimensions and proportions, viz. 1. square mètre (say $10\frac{1}{2}$ square feet) of kiln grating surface to every 180 cubic mètres (say 6180 cubic feet) of chamber space, and that on this kiln grating

surface should be burnt per 24 hours 270 kilos. (say 594 lbs.) of pyrites yielding 40 per cent. of sulphur, or 150 kilos. (say 330 lbs.) pyrites to every 100 cubic mètres (say 3432 cubic feet) chamber space, or in other words about 3.84 lbs. of sulphur ex-pyrites per 24 hours to every 100 cubic feet of chamber capacity.

Dust Trays.—In this country the dust is more commonly treated by introducing two cast-iron plates into each of a set of kilns such as shown in fig. 6. They may be 5 ft. 6 in. long, about 1 ft. 6 in. wide, and 1—1½ in. thick, resting in the brickwork at both ends, and along one side. The outside edge should have a flange or border 2—3 in. high. Separate doors should be provided opposite the trays, and these may be in two halves, to work easier on the hinges. The trays may be so fixed that on their requiring renewal, say every six months, they may be replaced without stopping the kilns. The dust may be charged with a scoop from the front, and raked over about 4 times every 12 hours. A hanger of wrought-iron should be suspended from the arch of the kiln, and pass under the front of the tray to prevent its sagging down with the great heat. This plan of burning the dust or smalls has the recommendation of Mr. McCulloch, whose great practical experience of sulphuric acid making certainly entitles him to be considered one of the highest authorities upon the subject. By this arrangement he found the dust after burning to contain only 1 per cent. more sulphur than the cinders of the lump ore.

Concerning kilns generally, he adds that they should have air-tight doors, and with a superficial area of 17.25 feet, or say 5 feet 2 inches $\times$ 3 feet 4 inches, they may be charged with 3¼ cwt. of pyrites per 12 hours, and ½ cwt. of dust to each tray alternately per 24 hours, thus the total charge per kiln would be 7½ cwt. per 24 hours. We quite agree with him in preferring to charge a little and often rather than introducing heavy charges at long intervals, and in the opinion that the extra air admitted does no harm.

For Gas-works Refuse.—Sometimes the refuse oxide of iron from gas-works may be obtained in such quantity as to be worth burning alone for the sake of its sulphur. Mr. Hills has invented a peculiar kiln for roasting this material, which consists of fire-brick furnaces provided with a series of shelves composed of fire-tiles, leaving a space of five or six inches between the shelves. The sulphur oxide of iron is placed on these shelves in thin layers. The shelves are connected alternately, at back and front, by apertures, and the gas from the lower shelves passes by a side descending flue, to the back of the furnace, where it rises by another side flue, to enter the third shelf. Air is admitted, as may be found necessary, at the ends of the shelves by sliding doors.

Re-burning Waste Pyrites.—Of the kilns already described there is none which will extract all the sulphur contained in an ore in one roasting: generally about 2 to 6 and even $7\frac{1}{2}$ per cent. remains unburnt. Sometimes the largest pieces found among the cinders are dressed and put through the kilns a second time on shelves or other contrivances, so that they shall be acted upon by the heat evolved from the burning raw ore (as with the dust trays); in other cases alternate charges are made of raw and burnt ore, but either method entails great expenditure for labour.

Sulphur lost in reducing Metallic Sulphides.—It may be said of almost all metallic sulphides that previous to extracting the metals from them, it is necessary to expel the sulphur which they contain.

This is too often done without any regard to the economization of the resulting sulphurous oxide which may be conveniently applied to the manufacture of sulphuric acid, and in most instances the noxious but valuable gases are left to find their way into the atmosphere, to such an extent that the annual loss of sulphur in England in this way must amount to many thousands of tons.

Spence's Kiln.—Spence's patent kiln, now in fairly

extensive use, was invented to remedy this evil. The bed is formed of fire-brick, and so arranged that heated air from a furnace shall pass underneath it, through flues communicating with a chimney. The ore, previously reduced to a fine state, is spread on the bed, over which a brick arch is turned to form a chamber for the accommodation of atmospheric air, which is admitted at an aperture in the kiln wall and becomes sufficiently heated in its passage to oxidise the sulphides and thus liberate sulphurous acid gas. This is a gradual process. A portion of the ore is inserted at the end farthest from the furnace, and spread to a depth of 3—4 inches. It is passed along the bed a stage at a time by means of a flattened bar, introduced at side doors provided for the purpose, at stated intervals, the time being dependent upon the character of the ore under treatment, which should be entirely deprived of its sulphur by the time that it reaches the end nearest the furnace. These kilns may be made 100 feet long if necessary. Of course they consume a considerable amount of fuel.

Gerstenhoefer's Kiln.—Some fifteen years since, Moritz Gerstenhoefer, of Freiberg in Saxony, devised a kiln which should extract *all* the sulphur contained in a sulphide by means of the heat generated by the burning ore itself. The plan was tried at the Royal Saxon sulphuric acid works, and was so successful, that it is now in more common use all over the Continent than perhaps any other description of pyrites kiln.

Herr Gerstenhoefer claims that the principal advantages obtained by the use of his invention are the following:—

1. The ores are operated upon in a very divided state, so as to expose a large surface to the action of the air, and this surface is always being renewed.
2. The air which must be admitted is forced to come perfectly into contact with the ores.
3. The ores are brought into the kiln by mechanical means continuously in such quantities as may be required, so as to maintain a continual roasting process.
4. The air

or draught is also brought into the kiln by mechanical means, in order to be able to regulate the draught according to the quantities of ore, and also to prevent the influence of storms, which, he says, are sometimes very troublesome in working with grate kilns. 5. The heat produced by the ores is not led away uselessly by the gases, but is employed to heat the new ores and the air.

Figure 12 shows a front view of this kiln and a vertical section.

In the drawing the furnace is shown in connexion with other similar furnaces. After drying the ores the pulverized pyrites are put into the feed-boxes, *a*, above. These feed-boxes are furnished with coarse wires or sieves, *b*, to keep back stones, lumps, and large pieces, in order to prevent them from breaking the feeding rollers. The bottoms of these feed-boxes are funnel-shaped, and are divided into three compartments. The pulverized pyrites fall through the boxes on to feeding rollers, which are furnished each with six ribs, and are the length of the boxes. The rollers are made to rotate slowly, and conduct the pulverized pyrites down to the vertical openings, or slits, *d*, through which they fall down into the main chamber of the furnace. The inclined iron plates placed above the feeding rollers can be taken away in order to clean the slits if necessary. The feeding rollers are actuated by screw wheels, *e*, and the screws, *e'*, which are turned by any suitable moving power. The stamped ores fall down through the slits, *d*, on to the upper three ore bearers, *f*, from whence after the pyrites have reached a certain angle they will fall down on both sides of the bearers on to the bearers below. The ore bearers are made of fire-clay, their form being prismatic, as will be seen in the figures. As the pyrites are fed in continually from above, the pulverized substance will keep falling on to the bearers below, and will pass from one to the other set of bearers throughout the whole chamber down to the bottom of the oven. The form,

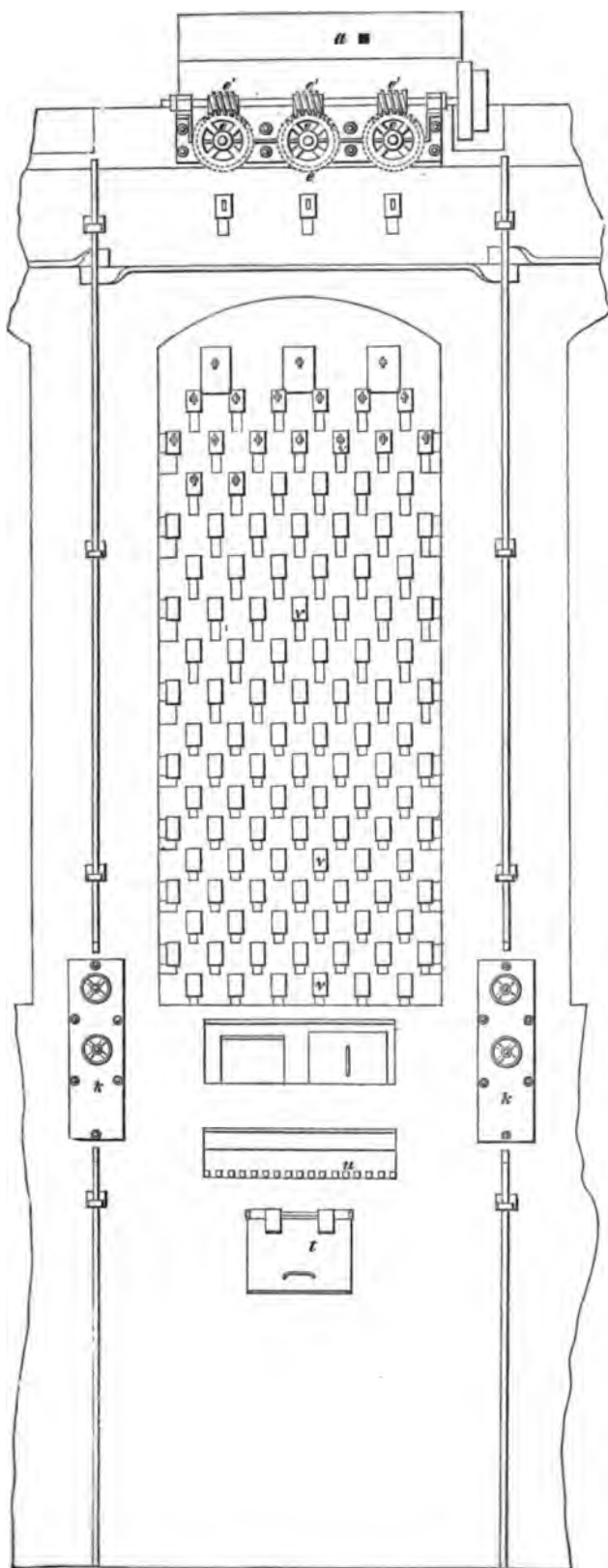


FIG. 12.

D

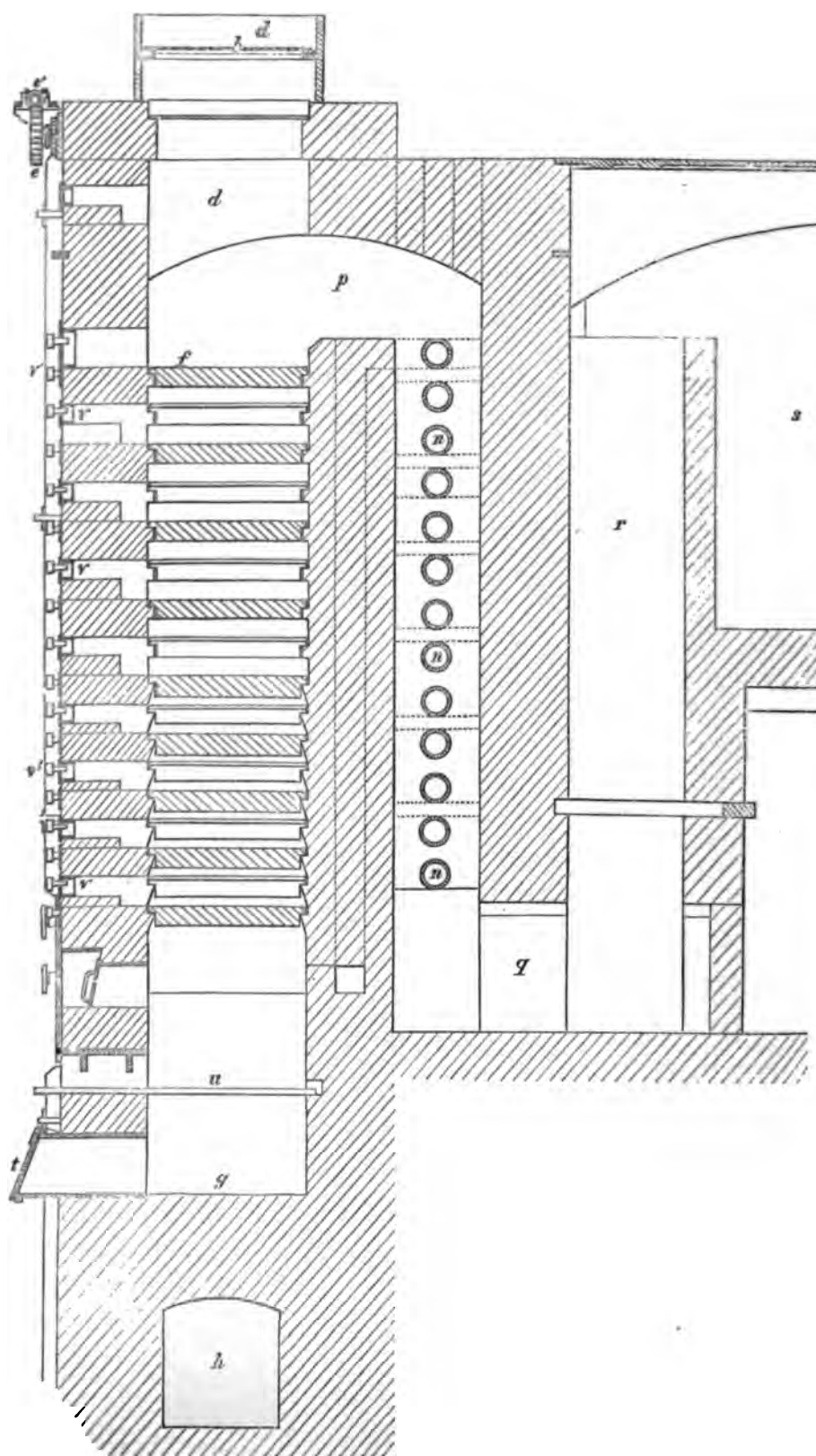


FIG. 12. Vertical Section.

situation, and arrangement of the bearers, will produce a great distribution of the pulverized ore, so that a large amount of surface will be exposed, and the roasting of the ore will proceed, during its gradual passage down from top to bottom, while the passing air is in constant motion in the opposite direction. Again, the form and situation of the bearers will be the best for the perfect employment of the heat, because the heat which is produced by the burning ores will be taken up by the passing draught, and will be reflected to the bottom surface of the next upper bearer, from whence it will be reflected again to the pyrites immediately below. The draught which is necessary for the roasting process must be produced by mechanical means, either by means of blowing engines or ventilators, partly because at some parts of the apparatus the heated air must be forced to move downward, partly because the quantity of the air can be more easily measured and regulated if admitted by mechanical means. The blowing engines will force the air into the main air channel, *h*, which runs below all the furnaces, from whence it will pass through a channel into the separate ovens. The air is then conducted to the wind-boxes, *k*, from whence it will find its way by the valves into channels. One of the channels leads by the shortest way into the lower parts of the furnace, and is only to be used if too high a temperature in the oven requires an admission of cold air. The other channel leads the draught in the clay pipes, *n*, *n*, and it is forced to pass through them from below to above one after the other. From the top pipe the draught is forced to go downward through a channel in the middle wall, and terminates in the lower part of the oven below the ore bearers. In the oven itself the draught must find its way through the ore bearers, and here it will come into intimate contact with the ores on the bearers, or with the falling ores, whereby it will increase its temperature and take up the sulphurous acid, and when the gases arrive at

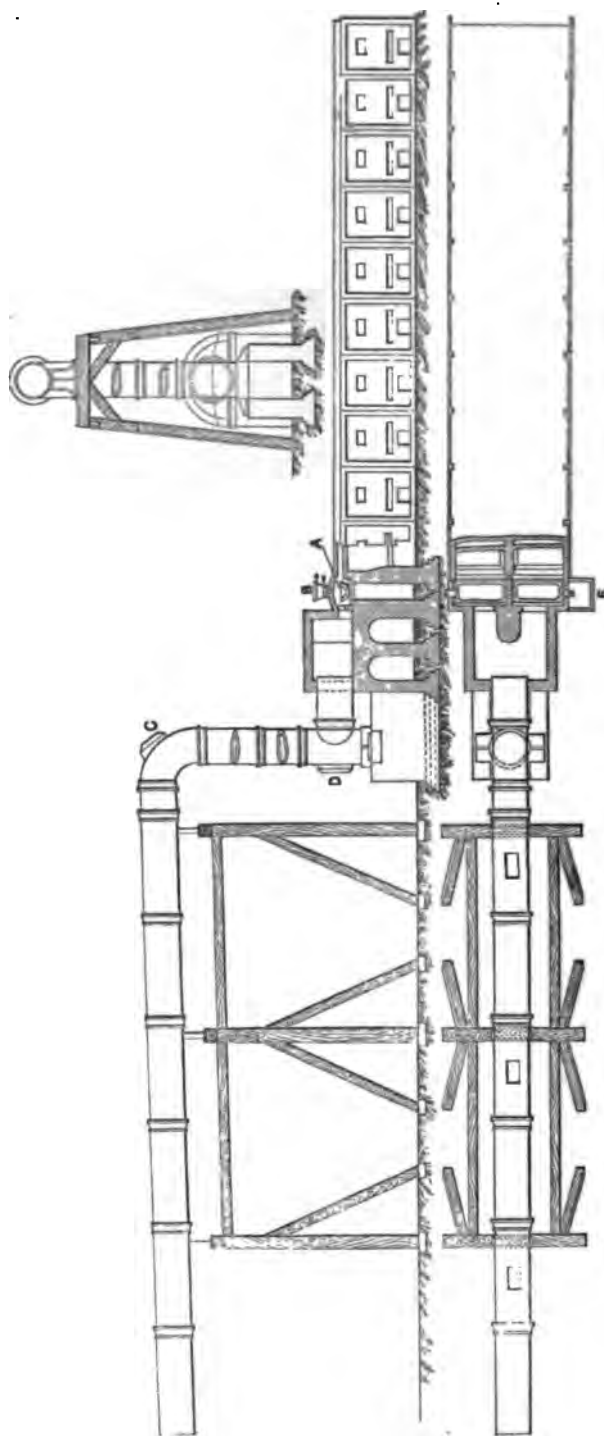
the upper part of the oven, they will be perfectly ready for the process of conversion in the lead chambers. From this upper part of the oven the gases pass off through the wide opening, *p*, into the back part of the oven, where they are forced to go downward, and to deliver their heat to the clay pipes, *n*, through which the fresh air is admitted as already mentioned. The warming of the fresh air will thus be very effectually performed as it passes along, as it goes in the opposite direction to the sulphurous gases. The gases will escape through the opening, *q*, at the lower part of the oven, and through the chimney, *r*, into the wide chamber, *s*, which is intended to give an opportunity to the gases before being brought into the lead chambers to deposit all the dust of roasted ores, and also the arsenious acid that may have been carried off from the oven. This chamber is provided with cast-iron plates on which the pulverized pyrites are spread out to dry, the pyrites being sometimes delivered in a wet state from the mining works. The roasted ores are brought out of the oven through the tight-fitting door, *t*, which is in so high a position that the ores may come directly into carriages or trams placed on rails, and may be withdrawn in this manner. There is a broad slit left open in the front wall of the oven above the door, *t*, in order to put grate bars, *u*, *u*, through it to receive fuel to set the oven on fire on commencing the operation. The front wall of the oven is built in such a manner that there are left openings, *v*, *v*, in it between every set of ore bearers; these openings are to be shut up, either by loose stones or by iron boxes with plugs, *v'*, in order to be able to reach every single bearer, and to clean it, if necessary, by simply withdrawing the stones or plugs and inserting a tool for the purpose. The interior of the oven is built of fire-clay bricks, and some of them being of peculiar form must be made on purpose. On all parts of the oven where a choking may happen by accumulation of dust, the clearing out of the dust is to be done through doors or openings made by removing the loose stones or bricks.

In beginning the roasting process, the oven is set on fire by wood or other suitable fuel placed on the grate bars, *u, u*, and afterwards the heat is increased by supplying coke until there is a red heat all throughout the oven. The grate bars, *u, u*, are then taken away, and the slit through which they are withdrawn is shut up, and the blowing engine and feeding apparatus are started. The roasting process will begin immediately, and will always continue uninterruptedly as long as pyrites are supplied and the oven is kept from choking. It will only be necessary to take care that the falling down of the pyrites from one set of ore bearers to the other be maintained, and any improper accumulation of pyrites on the bearers is to be prevented by means of a thin iron hook, with which the bearers are to be cleaned from time to time through the openings, *v, v*, in the front wall. If very clean ores, rich in sulphur, be operated upon, a cleaving together of the sulphuret on the bearers will take place on account of the high temperature; this is to be prevented by mixing the ores with roasted ores in order to diminish the temperature.

Economizing Heat from Kilns.—In some manufactories the heat given out by the kilns is economized by placing pans over them in which acid, for example, can be partially heated before concentration in another vessel, but we cannot approve of this plan, for the economy effected is very small compared with the great risk that is run of a small leak in the pan destroying the brickwork of the kilns, and necessitating the stoppage of the whole manufacture, whilst the costly repairs entailed are being done.

Daglish's large Nitre Oven.—By the kind courtesy of Messrs. R. Daglish and Co., St. Helen's Foundry, Lancashire, we are enabled to give the following views and particulars of their improved nitre oven, which may be used with great advantages over the old-fashioned plan in cases where the manufactory is on a sufficiently extensive scale.

Figure 13 shows plan, elevation, and sections of the



SCALE 1/4 INCH TO 1 FOOT.

FIG. 13.

arrangement. *A*, the cast-iron nitre pot is stationary in the kiln, and is filled with nitre through a cast-iron hopper; *B*, the gas is prevented escaping by means of a sliding hopper; some firms use two such slides. The acid used must be made warm before putting it into the pot. This is effected by keeping a supply in a lead-lined iron cistern on the top of the kilns, whence it is run into the nitre pot through a luted lead pipe, thus,—



There are manholes at the elbows of the long iron pipe, *C*, *D*, to admit of the cleaning out of the deposit of sulphate of iron, arsenic, &c., which accumulates there in a considerable degree. The nitre pot is generally put over the end kiln of each block, so that the waste heat of the burning ore may boil the pot and generate the nitric acid gas. As a rule 5 lbs. of nitre are added to 1 cwt. of Mason's (Spanish) pyrites, which contain about 49 per cent. of sulphur. To 56 lbs. of nitre are added $2\frac{1}{2}$ to 3 gallons of sulphuric acid from 116° to 130° , Twaddell. This acid is not added all at once, but, say, every quarter of an hour, in this way a *continuous* stream of nitric acid gas is produced, instead of the intermittent flow which is the result of the ordinary method of charging, and the sulphate of soda residue is prevented from getting thick. When the charge of nitre has been in the pot about two hours, the plug is withdrawn, and the sulphate of soda is run out on to an iron plate, *E*; if it be too thick to run, the plug must be replaced for a short time and a little more acid added.

CHAPTER II.

CHAMBERS.

EVEN at this late stage in the history and practice of sulphuric acid making, manufacturers are not yet agreed as to the best form or arrangement of chambers, and some of the plans most recently proposed, by men too of scientific attainments, are merely modifications of the earliest suggested shapes, and possessing the same faults and objections.

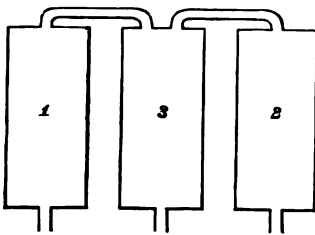
Capacity, Shape, and Arrangement.—It has generally been found that more satisfactory results are obtained from a large capacity than from a small one, particularly in the matter of reducing the consumption of nitre. Wagner calculates that when burning brimstone each 20 kilogrammes of sulphur burnt in 24 hours require 30 cubic metres capacity, and when pyrites are used a larger proportional space is necessary. The greater space demanded for the same amount of work when using pyrites is owing to the fact that the residual nitrogen is much greater in this case, the iron of the pyrites requiring nearly half as much air for oxidation as the sulphur with which it is combined.

As regards the shape for a chamber, a cube is the most economical form in reference to the quantity of lead employed to give a certain internal capacity, but in practice an oblong, or approach to a double cube, is commonly stated to work better. The width should exceed the height by one or two feet for convenience in building, which will be described presently. Probably no two manufacturers have chambers of the same proportions.

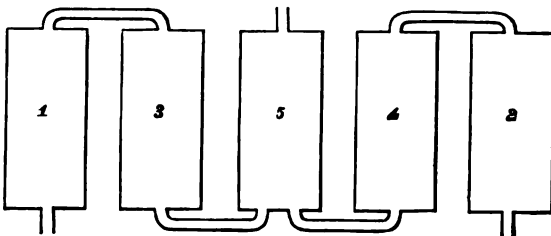
The following figures indicate how widely some of the older forms differed.

One at	Bradford	is	75 feet long by 35 feet wide by 35 feet high.
„	Newton Heath	is	75 „ 40 „ 40 „
„	Flint	is	140 „ $24\frac{1}{2}$ „ $19\frac{1}{2}$ „
			100 „ 20 „ 18 „
			120 „ 25 „ 20 „
„	Corkicle	is	60 „ 20 „ 18 „

These were all single chambers. Their average cubic capacity is about 66,000 cubic feet and the proportion of lead to capacity used in their construction varies from 12·86 per cent. to 23·11 per cent.



But also in the matter of number and disposition just as much variety exists. For instance, they may be arranged in a triplet; Nos. 1 and 2 being in connexion with the kilns, are called “working” chambers, and discharge their un-



condensed gases into No. 3, or “receiving” chamber, which communicates with a condenser or chimney.

Or they may be a quintet. Here 1 and 2 will be the “working” chambers, 3 and 4 the “receiving” chambers, and No. 5 will act as a condenser.

Succeeding the old-fashioned single chambers, which can rarely be found now-a-days, a plan which was for a time rather in favour consisted in allowing the gases from two separate sets of kilns to enter two disconnected chambers placed side by side, those portions of the gases which escaped condensation in these chambers being conducted into a third chamber, which thus formed a condenser common to the other two.

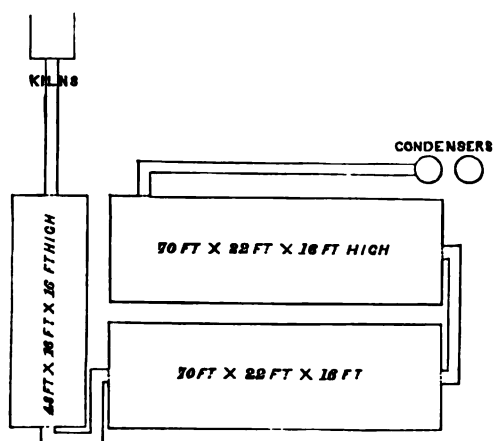


FIG. 14.

Figure 14 shows the arrangement and sizes of a set of chambers in the Eastern Counties. The total capacity of the three chambers, 61,568 cubic feet, would require about 15,632 square feet of lead for the chambers alone, or 25·39 per cent. of lead to space.

Another manufacturer has recommended the following disposition (fig. 15). Height of condensers 23 ft., diameter 4 ft. 6 in., bottom lead of trunk to turn up 6 in., chamber bottom lead to turn up all round 6 in., chambers to be of 5 lb. lead, trunk 7 lb., condensers and connexions 10 lbs.

Foreign Plan.—In figure 16 (frontispiece) is seen the plan adopted by a Continental manufacturer.

1 2 3 4 are the first, second, third and fourth chambers.

A B C D E (first-floor plan) are places where samples of acid are taken to show the progress of the manufacture.

F G (ground plan and long. section) are large leaden gas-pipes

between the chambers 3 and 4; arrows mark the course of the gas. The gas descends from chamber 4 into *H* (ground plan and long. section) a leaden or earthenware reservoir where it is divided, half going through one set of jars and

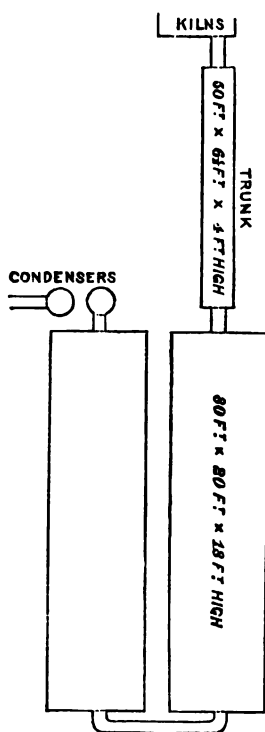


FIG. 15.

half through the other. Arrived at J, the gas is carried underground in drain-pipes to the main chimney.

K (long. sect.) a wooden box lined with lead and connected with each chamber by means of leaden pipes [presumably for the purpose of supplying water to cover the chamber floors at starting].

L (first-floor plan and trans. sect.) entrance doors to chambers.

M (first-floor plan) leaden pipes through which the acid runs from chambers 1 to 2, 2 to 3, and 4 to 3.

N (first-floor plan and long. sect.) steam pipes.

P (ground plan and both sect.) stoneware jars and connecting pipes for reducing the escaping gases.

Q (ground plan and trans. sect.) leaden channels under the jars to conduct the acid.

Sections *E F*, *G H*, and *I K* fully explain the construction of the kilns, nitre pot oven, and evaporating pan.

The joists which support the top lead of chamber 3 measure 9 in. by $4\frac{1}{2}$ in., and have no support except at each end. The lead of the chambers is 6 lbs. per square foot, and that for the connecting pipes is 9 lbs. per foot. The lead pipe connecting chambers 1 and 2 is situated very near the tops of the chambers. The cast-iron pipe leading from the kilns to chamber 1 is $1\frac{1}{2}$ to 2 in. thick, and the plans show it to be about 2 feet in diameter. It does not reach to chamber 1 by 3 or 4 feet, being connected with chamber 1 by a piece of lead pipe of that length, having about the same diameter as the iron pipe and made of lead $\frac{1}{2}$ in. thick. The chambers have no internal fittings, nor any apparatus for denitrifying. The apparatus underneath the chambers does not denitrify, but the weak acid placed in the jars absorbs the gases. The main steam pipes are of copper, 2 in. diameter, from which lead pipes 1 in. diameter conduct the steam to the chambers. The cocks on the steam pipes are of lead mixed with some other metal; they are made in France. The specific gravity of the acid in chambers 1 and 2 is 1.524; in chamber 3, 1.566; in

chamber 4, 1.315; in the jars it varies between 1.076 and 1.21, according to how long it remains in the jars before it is carried up into chamber 3. Each jar has a hole in it near the bottom, in which a cork is inserted before putting some water or weak acid into them. This cork is taken out by the aid of a pointed thin strip of iron every time it is desired to empty the jars. Underneath the holes there is laid down a leaden channel, down which the acid runs into a leaden reservoir, from which it is carried up into the large chamber (No. 3). The pressure of steam is kept at about 48 lbs. Acid is heated and evaporated a little in leaden pans on the top of the kilns and is then run into other evaporating pans preparatory to being concentrated in a platinum retort.

In this manufactory 8 lbs. of nitrate of soda are used to every 100 lbs. of sulphur in the ore, which is Norwegian, containing about 45 per cent. As 6—7 per cent. of sulphur remains in the ore after burning, the actual consumption of nitre is almost $9\frac{1}{2}$ per cent.

Mr. Smith's Remarks.—Mr. H. A. Smith has made many inquiries into the best form of leaden chamber, and the results of his observations point, he says, to one of about the following dimensions: length 150 feet, width 25 or 30 feet, height 10 or 12 feet. "There is thus a large condensing surface, the mixed gases coming readily in contact with all parts of the chamber, whilst they are also in contact with the previously condensed acid which rests on the sides of the chamber."

Mr. Smith further remarks that "*the action between the gases is very trifling towards the top of the chamber, the greatest amount of action being at the bottom. This leads me to the conclusion that the upper portion of the chamber acts merely as a reservoir for the gases, which it supplies, as they are wanted, to the lower portions, where the conditions are more favourable to the formation of acid. If, now, instead of using the chambers as at present employed,*

the height were to be lessened, there would then be a much greater condensing surface than in the present form. And here I should just like to say a word respecting condensing surface. *It seems to be tacitly admitted among the greater proportion of sulphuric acid manufacturers that the one thing needful for complete condensation is 'condensing space;'* and 'condensing space' and 'condensing surface' are often, indeed generally, used as synonymous terms. I have shown that very little, if any, action takes place where there is no surface; the presence of some tangible substance seems to influence in some indirect way the action of the gases upon each other. This also seems to be the common-sense view to take, but even among thoroughly practical men the confusion regarding 'space' and 'surface' seems to be very great."

He continues:—"Suppose the height of the chamber were diminished, the condensing surface is increased, and there is nothing admitted into the chamber which would tend to baffle the draught in any way—indeed, according to the shape of the proposed chamber, the draught will be rather increased; we also bring the gases more in contact with any already-formed acid at the bottom of the chamber, which I have shown determines very rapidly the action between the gases. This form of chamber also would be much more easily managed than the one now in use, which, from its very unwieldiness, involves the manufacturer in many expenses which are in reality perfectly needless. If again the mere item of expense be considered, I think every one will agree that the expense of building a chamber half the size of that in use at present would be a very considerable advantage to the manufacturer. The dimensions of the chamber I would recommend are as follows:—

Length, 160, 200, or even 210 ft.

Width, 30 ft., increasing in ratio to length.

Height, 10 ft., and for any length of chamber the height should not be increased."¹

¹ Dr. Schwarzenberg has published the results of some experiments

Let us now quote a few words from another authority on the subject, Mr. Muspratt.

“Notwithstanding the number of years that sulphuric acid has been, it may be said, a staple manufacture in England, and so much opportunity has existed for studying this process, much misconception as to the true action of chambers exists. The forms, shapes, and other matters in connexion with them which have been proposed and built almost exceeds belief—their name is legion; but the prevailing idea, which appears to have actuated many inventors of these ingenious constructions, appears to have been that the action of condensation in a chamber was simply that of a distillation process; that as the vapour from a boiling liquid in a still or retort condenses on the cold surface of the worm or other refrigerating apparatus, so with a sulphuric acid chamber, the cold walls condensed the acid vapour contained inside. Evidently with this idea in view, chambers have been built resembling flues; these, having a large surface of cold lead, were supposed to favour the production of the acid by condensing it immediately on its formation. It is related that a clever chemist across the channel constructed a most novel sulphuric acid apparatus. It consisted of an almost fabulous length of lead tubes coiled round and round an apartment like a gigantic still-worm; that sulphurous acid and nitrous gas were introduced at the highest end; these reacted on each other, the sulphuric acid when formed passing the whole length of the tube, a constant stream issuing from the other end.

“It was not stated what length of time this system continued, but certainly on the face of it not long.”

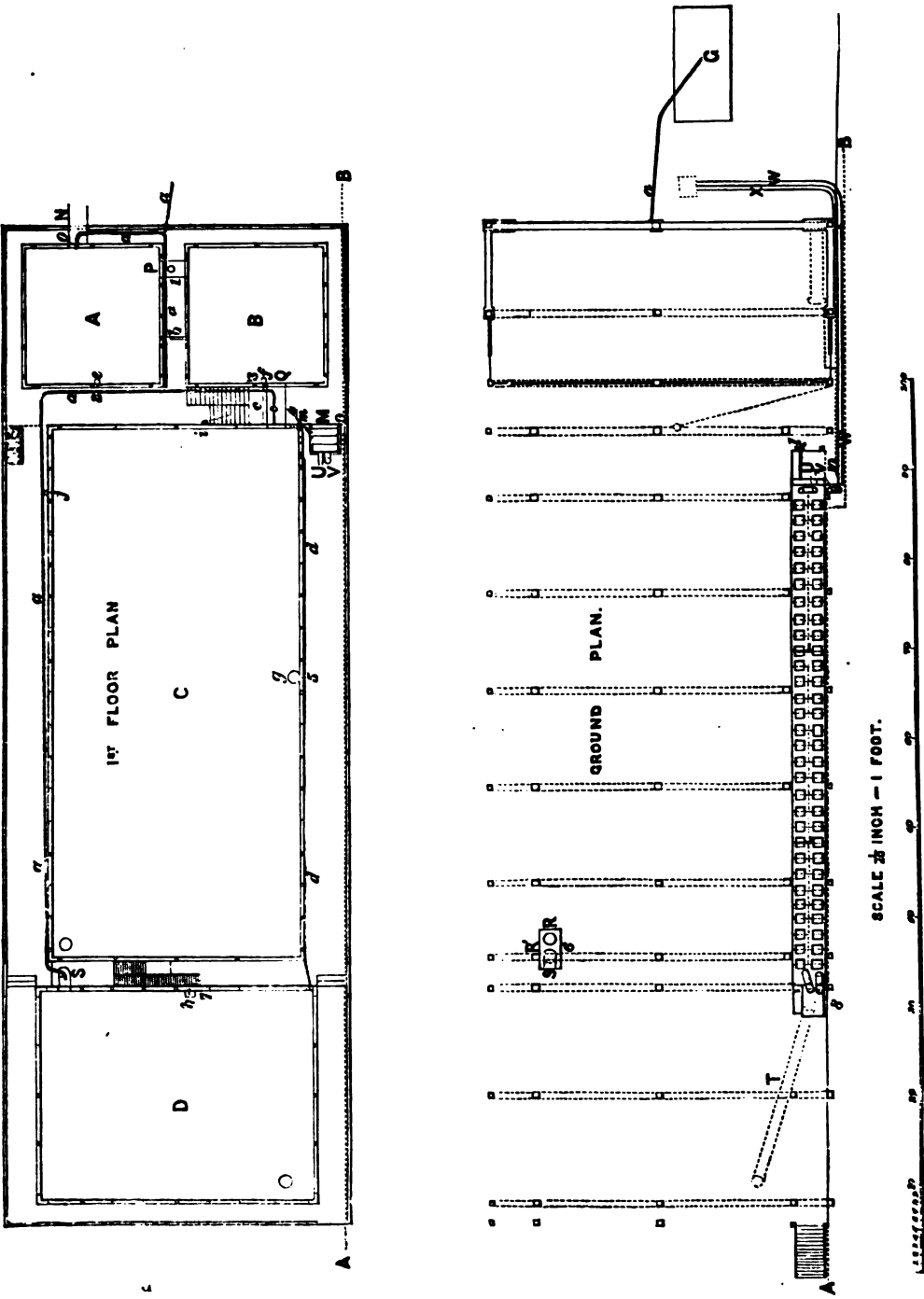
which tend to show that the gases spread themselves along under the roof of each chamber and thence descend, condensing into tiny spray as they fall, and he found that even when these gases were admitted near the bottom of a chamber that they immediately rose to its roof, there spread out and condensed. This seems to us to strengthen the impression that height is necessary and that space is as much a desideratum as surface.

Is surface, then, become more necessary since Mr. Muspratt wrote his work?

We beg to call the reader's special attention to those parts of Mr. Smith's observations which we have put in italics, and also to the following deductions from his figures. A chamber measuring 210 ft. $\times$ 30 ft. $\times$ 10 ft. high will require 17,400 square feet of lead for the chamber alone to produce a cubic capacity of 63,000 feet, or in other words, the lead required is *27.62 per cent. of the capacity*. One having the dimensions 150 ft. $\times$ 30 ft. $\times$ 10 ft. high will require *28.00 per cent. of lead to capacity*. One 420 ft. $\times$ 60 ft. $\times$ 10 ft. will take 60,000 square feet of lead for 252,000 cubic feet capacity, or *23.81 per cent.*; but this last is an almost *impossible shape to erect in practice as no timber joist could be got to lie horizontally (without sagging down) in a span of 60 ft.*

Another important point is the quantity of acid which must (or should) always remain in the chambers. A "cup syphon" will not run well with less than 2 inches, and nothing will run with much less than an average inch, and indeed an inch may be calculated as the lowest level possible to be maintained, for a sufficiency must always remain to "lute" the chamber or prevent the escape of gas, and no one can calculate, in practice, upon having his chambers built with such mathematical accuracy and precise workmanship, that he could be certain that less than an inch of acid would suffice to cover every part of the bottom and "lute" the sides. A chamber 210 ft. by 30 ft., having one inch of acid all over the bottom, will necessitate the locking up of 525 cubic feet of acid, which, at 100 lbs. per cubic foot, is over 23 tons; and a chamber 420 by 60 would always have a dead weight of over 46 tons lying in it unproductive.

A modification of the arrangement shown in the frontispiece was adopted by us. Fig. 17, will indicate at a glance the main points in which the constructions differ. Our



right is

FIG. 17.

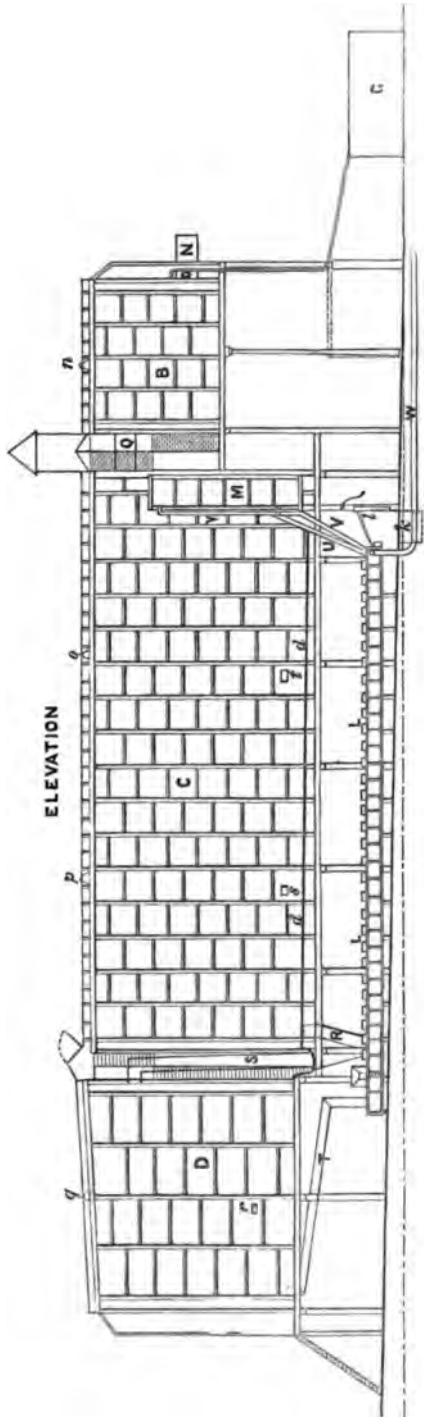


FIG. 17.

works being in the south of England, where neither hard frost nor heavy snowfall is prevalent, we felt no necessity for having a costly roof, and instead of this the tops of the chambers were made to incline 1 foot in 30, to throw off the rain. Owing to the direction of this slope, the elevation shows it only in chamber *D*, but the other chambers have an equal slope in the direction of the top joists.

<i>A B C D</i> , four leaden chambers.						Cubic ft. in.
<i>A</i> is 15 ft. long	by 15 ft. wide	by 13 ft. 6 in. average height				3037 6
<i>B</i> is 15	„ 15	„ 13 ft. 6 in.	„			3037 6
<i>C</i> is 60	„ 27	„ 23 ft. 6 in.	„			38,070 0
<i>D</i> is 30	„ 23	„ 22 ft.	„			15,180 0
Total capacity . . .						<u>59,325 0</u>

G Steam-boiler.

L (ground plan and elevation), leaden trunk for reducing the escaping chamber gas.

M (first-floor plan and elevation), coke condenser for same purpose.

N (first-floor plan and elevation), cast-iron pipe 22 in. internal diameter, leading from the kiln-stalk.

O (first-floor plan and elevation), leaden pipe, 25 in. internal diameter, connecting *N* with chamber *A*.

P (first-floor plan), leaden pipe connecting chambers *A* and *B*, 25 in. internal diameter, with a hole in the top, furnished with a luted cover and drip pipe underneath, to convey the acid to a suitable place for trying its strength.

Q (first-floor plan and elevation), leaden pipe connecting chambers *B* and *C*, same size, &c. as *P*.

R (ground plan and elevation), leaden pipe 19 in. internal diameter, descending from *C* into *R'*, a wooden box lined with lead.

S (elevation and both plans), leaden pipe 19 in. internal diameter, ascending from the box *R'* and entering *D*.

T (elevation and both plans), leaden pipe, 15 in. internal

diameter, descending from *D* into a box communicating with the beginning of *L*.

U (elevation and both plans)), leaden pipe, 8 in. internal diameter, ascending from *L* and entering *M*, near the bottom.

V (elevation and both plans), leaden pipe, 8 in. internal diameter, descending from near the top of *M* into

W (ground-plan and elevation), glazed stoneware pipes 9 in. internal diameter, conducting the escaping gases, after reduction, first into a lead-lined box, where the liquid arising from the last condensation of the gases may be caught, and leading thence to the main chimney.

a (first-floor plan), steam-pipes.

b (first-floor plan), small trough through which acid flows from *A* to *B*.

c (first-floor plan), leaden pipe conducting acid from *B* to *C*.

d (first-floor plan and elevation), trough conducting acid from *D* into almost the farther end of *C*.

e, f, g, h (first-floor plan), trays inside the chambers to catch the condensing gas, which then runs through a short pipe to the outside of the chamber, where it is caught in cups and its strength tried.

i (first-floor plan), syphon for running acid out of *C* into the evaporating pan.

j (first-floor plan), syphon for running acid out of *C* into tank for manure-making.

k (elevation), underground tank lined with lead, to hold water or weak acid.

l (elevation), leaden pipes for raising water or acid from *k* to *L*.

m (first-floor plan), pump for lifting acid out of *L* into *C*.

n, o, p, q (elevation), bell-glasses luted into the top leads of the chambers to admit light.

r, s, t (elevation), panes of glass let into the sides of the chambers to form windows, through which the colour of the gases may be seen.

Before commencing to work the chambers they must be luted, that is to say a sufficient quantity of acid (water is not so good for many reasons) must be put into them to prevent the gases escaping through the space intervening between the bottom lead and the lower edge of the side lead. In chamber *C*, the average depth was 1.9 in., in the others only just enough to lute them.

Course of the Gas.—We must now refer to the gases generated in the kilns. The description of the flue which brings these gases into the first chamber will be found under the head of construction, we now pass to the gases themselves. These enter chamber *A*, a little above the bottom, and a jet of steam is admitted just over their entrance. Thus mingled together they pass through the pipe *P*, high above the ground, into chamber *B*. Thence they enter chamber *C*, through the pipe *Q*, where a second jet of steam is introduced. After traversing the whole length of *C*, the gases descend through the pipe *R*, leading from the bottom of *C*, into a lead-lined box *R'*, whence they rise through the pipe *S* and enter *D* near the top, where steam is again admitted; passing out of *D* (at the opposite side from where they entered) through the pipe *T*, into the leaden trunk *L*, and thence to the chimney.

Chamber Day-book.—In order to regulate the supply of the various vapours (nitrous, sulphurous, and aqueous) and to control the reactions going on in the chambers, arrangements are made for obtaining tests of the strengths of the gases and acid at various points, and these are registered in a book called a Chamber day-book. The following extract of a day's working taken at random from our Chamber day-book will illustrate our meaning.

Date.	12 Noon.
1	142
2	122
3	147
4	134
5	142
6	154
7	121
8	103
A.B.	117
C.	115
D.	103
Depth	7.60
O.	40 cwt.
N.	136 lbs.
Used	for O.V. 0.30 „ Maure 1.40

Nos. 1 to 8 represent the different places at which the condensed chamber *gases* are caught in leaden cups for their strength to be tried by Twaddell's hydrometer.

1. Gases passing through the pipe *P*, connecting chambers *A* and *B*.

2 and 3. Gases collected at *e* and *f*, chambers *A* and *B* respectively.

4. Gases collected from pipe *Q*, between chambers *B* and *C*.

5. Gases collected at *g*, chamber *C*.

6. „ „ in the box *R'*.

7. „ „ at *h*, chamber *D*.

8. „ „ at entrance to trunk *L*.

AB. Overflow of acid from chambers *A* and *B* into *C*.

C. Acid in chamber *C*, tried from the syphon *i*.

D. Overflow of acid from chamber *D*, caught where it enters chamber *C*.

O. Pyrites or (brimstone) } Used per 24
N. Nitre } hours.

Depth is the depth made in chamber *C*, recorded in inches and tenths.

Air.—Every pound of sulphur burnt requires about 113 cubic feet of air for its conversion into sulphuric acid. This supply of air is regulated by the dampers on the kilns and between the final condenser and the chimney, or by a special contrivance which measures it at the same moment.

Steam.—It is as well to have steam at command in excess and under from 40 to 50 lbs. pressure per square inch. Super-heated steam is not so suitable. The quantity to be used may be roughly estimated as the equivalent of 2.5 to 3 parts of water for every 1 part of sulphur burnt. When too much steam is admitted the strength of the chamber acid is reduced; when too little, there is danger of chamber crystals forming. The increase of steam will correct

the evil. C. Rammelsberg says, "that when decomposed by water these crystals yield 22 per cent. oxide of nitrogen, 14 per cent. nitric acid, and 64 per cent. of nitrous acid." The appearance of the bell-glasses on the tops of the chambers as hereafter described, offers some guide; they should appear thoroughly moist inside. A green colour in the acid indicates a quantity of nitrogen peroxide in solution, generally due to want of steam. A self-registering steam-gauge affixed to the pipe conveying steam to the chambers, and placed where it cannot be meddled with, is a valuable safeguard. In fact too many precautions cannot be taken to ensure regularity and constancy in every department affecting the production of the acid. During the day things will go well enough, but the night workmen adopt every possible trick to evade their work.

Testing Chamber Gases.—Sulphurous gas has a pungent smell and whitish colour; nitrous gas has a sweet smell and a greenish-red colour.

Again. Take some of the acid caught on a drip tray (see hereafter) and add a little water. If the gas that arises looks sulphurous, too little nitre is being used; but if it smells very sweet and is heavy, especially if it rises red, too much nitre is being used. Acid containing much nitrous gas in solution will froth on being simply poured from one vessel to another.

Working Results.—The average working results of these four chambers were as follows:—

Ore burnt per day of 24 hours, 2 tons, containing 45 per cent. of sulphur, of which 6 per cent. were lost in the waste, giving 1747 lbs. of sulphur *burnt*, which produced a quantity of chamber acid at 113° Twaddell, equal to 2 tons, 5 cwt. 2 qtrs. 10 lbs. of "Oil of Vitriol" at 169° Twaddell (1.845 sp. gr.), that is to say, 292.27 parts for every 100 parts of sulphur *burnt*. The nitre used was 136 lbs. per 24 hours, or 7.78 per cent. of the sulphur burnt.

We cordially hail Mr. Smith's scientific little book, and

trust he will be encouraged to carry his inquiries still further, and to erect a chamber of the dimensions which he recommends, the result of whose working in practice we hope to hear in due course. In the meantime, as we are humbly striving to attain the same end with Mr. Smith—the *most economical* manner of making sulphuric acid—we venture to think he will not take it ill if we have made a few “odious comparisons” between his theory and our practice.

Temperature.—With regard to temperature; he advises about 200° Fahr., as the degree of heat best suited to the reactions in the chamber. “At this time, also [temperature 195° to 199°] the yield of vitriol obtained from this chamber was as nearly the theoretical amount as could practically be obtained; and it was found that whenever the temperature of the chamber was allowed to increase or diminish the result was bad.” The exact percentage obtained is not stated. Again he says, “The higher the temperature the more steam required.”

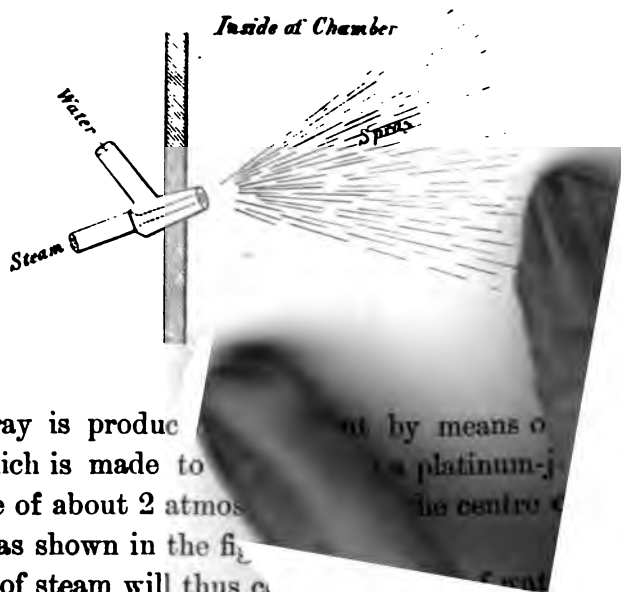
We never made observations upon the interior temperature of our chambers. The pressure of steam was kept at 40 to 45lbs. to the square inch.

The following extract from the *Chemical News*, Sept. 24th, 1875, is interesting:—

“Mr. Hermann Sprengel’s application of atomized liquids in operations where a liquid is made to act as an absorbent of a gas possesses great advantages. The method has been applied with success to the purification of coal-gas, and to the condensation of hydrochloric acid, and by its use great improvements in the production of sulphuric acid have been effected. It is well known that sulphuric acid as contained in the chambers contains about 50 per cent. of water, and that all this water was once steam, and was taken as such from the steam boiler. Before being condensed in the chambers this steam occupied a certain space, and moreover helped—on account of its heat—to expand

the bulk of the other gases used in the formation of sulphuric acid. In winter time the yield of acid is better and the consumption of nitre less, than in summer time and the greater the chamber space (i. e. the smaller the volume of gas allowed to pass the chambers in a certain time) the less will be the consumption of nitre (in proportion to the acid produced) and the easier will be the conversion of all sulphurous into sulphuric acid.

Hence, as the *lowering* of the temperature of a gas necessarily implied the *shrinking* of its volume, both of which favour the process of sulphuric-acid-making, Mr Sprengel commenced to manufacture sulphuric acid by means of what has been called '*pulverized or atomized water or spray*,' which he injects into the chambers as a substitute for steam. This effects (1) a saving of fuel equal to the amount which is required to convert this pulverized water into steam, and (2) a cooling of the chambers equal to the loss of the amount of heat which would have been generated by the combustion of the coal thus saved.



The spray is produced by means of steam, which is made to pass through a platinum-jet at a pressure of about 2 atmospheres. The centre of the jet is surrounded by water, as shown in the figure.

20 lbs. of steam will thus create a cloud-like mist, the actual weight

jet of the above size, amounts to about $\frac{1}{3}$ ton in 24 hours. These jets are placed in the sides of the chambers about 40 ft. apart. They are supplied with water from a tank above, while the steam is taken from the steam-pipes already existing between the chambers, or better from smaller ones put in their place.

At the works of the Lawes Chemical Manure Company the saving in coal amounts to about two-thirds of the quantity formerly burned. It is generally believed that a moderate temperature favours the formation of sulphuric acid, but Mr. Sprengel has found that the stronger the frost the better was the condition and the yield of the chambers.

The 'spray acid' has been produced with $6\frac{1}{2}$ per cent. less pyrites and with $14\frac{3}{4}$ per cent. less nitre than the 'steam acid,' which was made from the same material during the two years preceding the application of the spray. These numbers, moreover, refer to the yield of chambers without Gay-Lussac and Glover towers.

In factories where these towers are in use the saving will be probably one-third less, at least as far as steam is concerned. But as it is believed that a considerable proportion of nitrous acid becomes destroyed in the Glover tower by the heat of the gases from the kilns (i. e. broken up into oxygen and nitrogen) Mr. Sprengel thinks that for the sake of coolness this acid is better distributed in the interior of the chambers as spray. The Glover tower of course will still serve as an admirable instrument for concentrating chamber acid.

At the works of the Lawes Chemical Manure Company the construction of the apparatus came to about 10*l.* per chamber, while the savings in steam, acid, nitre, and labour during three months amounted to 5*s.* per ton of acid of 1.6 *sp. gr.*, made from pyrites.

No doubt different localities, different care, and different prices, will lead to different results. But even if the savings should elsewhere be considerably less, the result

will still appear acceptable, considering the simple and inexpensive means by which it has been attained and the large consumption of the article which it helps to cheapen, for 1s. per ton saved or wasted means, with reference to England's annual consumption, about 50,000*l.*"

Payen says the temperature may vary from 40° to 60°, but he forgets to give the scale: supposing it to be Centigrade, the relative figures by Fahrenheit will be 104° and 140°. Self-registering thermometers kept suspended in the chambers are very useful.

Other Plans.—A works erected a few years since in the north of England, and which was then considered of superior construction, was arranged in the following manner. There were 8 chambers, each 120 ft. long, 25 ft. wide, and 16 ft. high, arranged side by side in one line; supported on iron columns, and lined throughout with 5 lbs. lead. They were worked in two suites of 4 each and supplied from 2 sets of 18 kilns arranged underneath the chambers. The total capacity of the 8 chambers was therefore 384,000 cubic ft. Each kiln was charged with 6½ cwt. Tharsis copper pyrites per 12 hours. The dust pyrites was all burnt at another works in a special kiln provided with iron shelves. The pyrites contained 48—49 per cent. of sulphur and about 3 per cent. was lost in the cinders. Thus 468 cwt. of pyrites, divided among the 36 kilns at 6½ cwt. per charge, per 12 hours, was burnt in every 24 hours, and as this yielded 45 per cent. of sulphur (say 48—3), there were 23,580 lbs. of sulphur as sulphurous acid passing into the chambers in every 24 hours, or 16.28 cubic ft. of chamber space per lb. of sulphur converted per 24 hours. The yield was about 131 parts of monohydrated sulphuric acid for every 100 parts of pyrites charged, which, taken at 45 per cent. converted, is equal to 291 parts acid per 100 sulphur converted. The gases from the kilns were passed equally through two Glover's towers, and the gases leaving the chambers were taken collectively through a Gay-Lussac

column. These towers were arranged in close proximity to each other for convenience in attending to them, and stood at one side of the row of chambers.

The arrangement appears to be excellent, but there were three faults in the construction which call for some notice. The lead lining of the chambers was too thin, it should be rather 7 lbs. than 5 lbs.; placing the kilns *underneath* the chambers was a practice to be highly disapproved of on account of the risk of fire; and iron columns for supporting the chambers are very much inferior to stout timber standards, as the former cannot be strutted, and in case of anything going wrong with one, the extra weight thrown on the next is very likely to make it snap. They are also much more costly, under ordinary circumstances.

Mr. McCulloch's Plan.—Mr. McCulloch speaks highly of a plan of six chambers as worked by himself with very good results. These were 180 ft. $\times$ 21 ft. $\times$ 20 ft. high and were worked at 28 cubic ft. per lb. of sulphur per 24 hours, and gave 284 per cent. of oil of vitriol on the sulphur charged. The working chambers were never allowed to exceed 160° F., and the receiving chambers were similarly restricted to 90° F. The pyrites used at various times was 1st Norwegian (46.15 per cent. sulphur), 2nd Norwegian (38.17), Mason's (49.8), and Belgian (45.6), the amount of sulphur left in the cinders being respectively 3.5, 7.5, 3.5, and 2.5 per cent.

There were 40 kilns for the 6 chambers, divided into two 20's. The gas from the first 20 kilns was taken through a Glover tower, thence into chamber 1 and chamber 2, and then along a flue fitted with a damper, into chamber 5 and chamber 6, and thence into the absorber column. Two of these absorbers were provided; in case of repairs being necessary to one, the works could thus still be carried on. The gas from the 2nd 20 kilns was similarly taken first through a Glover tower, then into chamber 3 and chamber 4, and, as the others, through chambers 5 and 6 and the

absorbing tower. This arrangement was therefore equal to working two sets of 4 chambers each. Either set of 4 can be worked by closing the dampers, and also an extra charge can be put into the 20 kilns if necessary. The draught should be so that the gas will either come out or go in when the kiln doors are opened. The chamber drips were kept at 130° — 135° Tw. for the strongest, and 100° Tw. for the weakest.

The steam was never high pressed, and was best kept at about 11 lbs. per square inch.

A similar set of chambers provided with steam denitrating towers required only 3.5 per cent. of nitrate of soda.

French Plans.—Dr. Quesneville, before quoted, states that the number of chambers worked in a set in France is generally three, of which one is large and the other two smaller. He thinks the large ones should be 45 m. $\times$ 8 m. $\times$ 6 m. high, or say $146\frac{1}{2}$ ft. $\times$ 26 ft. $\times$ $19\frac{1}{2}$ ft. high, and he reckons the total cubic capacity of the chambers at 4000 cubic mètres, or about 137,312 cubic feet. He disapproves of the practice of exposing the sides of the chambers to the weather, as the working is much interfered with thereby. Finally the cost per 1000 kilos. of acid at 50° (? $B^{\circ}=107^{\circ}$ Tw.) is stated at 36.9 francs, or say 30s. per ton.

German Plans.—The dimensions recommended by Hasenclever as being the most economical are 40 m. long, 10 m. wide, and 10 m. high, or say 130 ft. $\times$ $32\frac{1}{2}$ ft. $\times$ $32\frac{1}{2}$ ft., and the quantity of sheet lead required will be only .45 square mètres per cubic mètre chamber capacity.

Dr. Affleck remarks that though a cubic is the most economical form for a chamber as regards the proportion of sheet lead required to produce a given capacity of chamber interior, this shape is not found to yield good results in working, and he explains the reason of this to be the simple fact that the production of sulphuric acid in the chamber is a continuous process, which, rapid at first when the gases meet each other in a condition of great

vitality, becomes much tardier as their strength wanes. Thus towards the end of the reaction the gases must be kept in contact with each other for a length of time in order to give them every opportunity for attacking each other, and therefore a cubical chamber is not so effective as an oblong.

With regard to the capacity of the chambers, he thinks that 3000—4000 cubic feet should be reckoned for every ton of pyrites burnt weekly.

As to the disposition of a set of chambers (for of course no one would think of erecting a single chamber now-a-days) he advises that $\frac{1}{2}$ or even $\frac{2}{3}$ of the total chamber capacity should be divided equally among two large chambers receiving the gases from the kilns, and that the remainder of the total capacity should be split up among three or four narrow chambers. In erecting chambers to take the gas produced by the combustion of 70 tons of pyrites weekly, and allowing 3200 cubic feet per weekly ton of pyrites, he would dispose the chambers thus:—

2 Chambers each 60 ft. × 50 ft. × 20 ft. high	} Total cubic capacity,
4 „ „ 60 ft. × 20 ft. × 20 ft. „	
	216,000 ft.

The total superficial area of these chambers would consume 43,200 square feet of lead, or $\frac{1}{5}$ square foot of lead per cubic foot of capacity. That would be 10,800 square feet of lead less than with the proportion of $\frac{1}{4}$ ft. per foot, which at 7 lbs. per foot is equal to 34 tons, no inconsiderable item. The doctor questions whether it would not be as well to put this weight of lead saved into the shape of extra thickness and thus make the chamber lives half as long again. He considers the best length to be 80—90 feet, and if much longer steam must be introduced at the sides as well as at the ends.

Concerning the number of chambers, Dr. Affleck is of opinion that they should not be fewer than 5 or 6, and that even 7 or 8 may be worked in conjunction with good effect

as regards utilizing all the gas. Twin sets of chambers also he approves of, and in that case the gases may be made to come from two separate sets of burners or kilns, and to pass first through, say, 2 chambers each, and then jointly through 3 or 4 more common to both. He thinks this will also tend to compensate irregularities.

In order to give a better idea of the main points on which the above quoted authorities agree or differ, we have put their figures into the form of a little table to which we have added the dimensions of a works in the Eastern counties and some details of a set of chambers worked by us.

In this comparative table we have not given any details concerning consumption of nitrate of soda, as that depends so much upon the arrangements provided for economizing the salts of which we possessed no specific information regarding each system respectively.

The capacity provided and sulphuric acid produced we have thought it well to proportion to the amount of sulphur *converted* and not to the amount *charged*, for this reason, that the work done by the chambers bears no relation whatever to the quantity of sulphur put *into* the kilns, but to the quantity of sulphur issuing *from* the kilns in the shape of sulphurous gases.

From the table we see that of the large sets of chambers viz. Mr. McCulloch's, Dr. Affleck's, and the octavo systems, the first named has much the largest capacity with almost the least consumption of lead, and that by burning a proportionally small amount of sulphur to the cubic foot of space the greatest production of acid is obtained. On the whole, this system of Mr. McCulloch's seems to leave little to be desired in the way of improvement.

The second of the large plans referred to, Dr. Affleck's, consumes the least proportion of lead, and is calculated to take more sulphur per foot, but we have no figures concerning the produce obtained. It appears to us that the small percentage of lead saved over the first-named system has

System.	No. of Chambers.	Dimensions of Chambers. Length Width Height.	Capacity in cub. ft.	Sq. ft. lead used.	Proportion lead to capacity per cent.	Sulphur burnt per 100 cu. ft. in lbs.	Acid produced per 100 sulphur burnt (not charged).	Remarks.
McCulloch	6	180 ft. × 21 × 20	453,600	93,600 ¹	20.63	3.43 ²	295.83	¹ No allowance is made in any case for turn over, turn up, lap, nor strap of any kind.
—	8	120 ft. × 25 × 16	384,000	85,120	22.16	6.14	291.00	
Smith	1	210 ft. × 30 × 10	63,000	17,400	27.62	—	—	
Hasenclever	1	130 ft. × 32½ × 32½ ³	137,312	19,012	13.85	—	—	² Reckoning his pyrites as having 4% of sulphur left in.
French	3	146¼ ft. × 26 × 19½ ³ and two others.	137,312	—	—	3.84 ⁴	—	³ Taking the mètre at 39 in.
E. Counties	3	70 ft. × 22 × 16 70 ft. × 22 × 16 48 ft. × 16 × 16	61,568	15,632	25.39	—	—	⁴ Taking 10 kilos. at 22 lbs.
Dr. Affleck	6	2/60 ft. × 50 × 20 ² 4/60 ft. × 20 × 20 ⁴	216,000	43,200	20.00	4.00 ⁵	—	⁵ Reckoning 3200 cubic feet per weekly ton of pyrites, and that 40% of sulphur is actually burnt.
Authors'	4	60 ft. × 27 × 23½ 30 ft. × 23 × 22 2/15 ft. × 15 × 13½ ²	59,325	13,696	23.08	2.94	292.27	

only been got at the cost of a width of chamber which is open to much objection. Fifty feet is a very wide span for a joist, and even supposing it to be always feasible to obtain timber of that length suitable for the purpose, which is certainly open to doubt, the joists would be extremely heavy and cumbersome, and would undoubtedly sink down in the middle very considerably. These drawbacks, are, we think, quite sufficient to counterbalance the small saving of lead, and only exceedingly good working results would repay such unusual proportions. The smaller chambers of this system, too, are liable to cause inconvenience in construction, by being exactly the same height as width, thus preventing the raising of the side sheets in the easiest manner.

The French system calls for no special remark, as we have not sufficient details concerning it.

Touching Mr. Smith's theoretical chamber, one of whose greatest objects was to be the economy of lead,—it will be seen that it consumes far more lead than any other in the table, more than twice as much as one plan given. Of course if it will do twice as much work as the same capacity put into any other shape, it is an improvement, but will it?

Hasenclever succeeds beyond doubt in effecting very remarkable economy in lead, but also at the expense of a most unwieldy shape, which we have no *à priori* reason for expecting to give improved working results. The height is greater than we have ever heard of before, and for no apparent end. It is in fact interesting to compare the extremes to which theory goes. Mr. Smith tells us that everything above 10 feet in height is so much space wasted, while Hasenclever advocates nothing under $32\frac{1}{2}$ feet. Dr. Affleck's largest chambers have only one-third the length of Mr. McCulloch's, but are twice as wide.

Concerning our own quartet we venture to think that they show as favourably as can be demanded of any small arrangement of chambers, though not to be compared with larger systems perhaps.

We learn from a journal of recent date, that an important improvement in the manufacture of sulphuric acid has been effected by Monsieur Fournet, at Bordeaux. By causing the gases from the kilns to circulate more than once in tubes filled with coke so as to form a homogeneous mixture, he has succeeded with a chamber space not exceeding 10,800 cubic ft. in burning an average of 1000 lbs. of sulphur, or say 9.26 lbs. per 100 cubic ft., obtaining 3000 lbs. monohydrated acid. If the sulphur is derived from pyrites the results are unusually high.

In some works it is the practice to convey the nitrous fumes into the chambers by channels separate from those conducting the sulphurous gas, but we fail to see what advantage arises from it.

Without question, the most common manner of introducing the oxides of nitrogen into the chamber process, in this country at least, is that already described, viz:—digesting nitrate of soda with sulphuric acid in iron pots in the sulphur kilns, and allowing the mixed gases to enter the chambers in company.

Burnard's Patent.—An important modification of this plan, however, has been made the subject of a patent by Mr. Burnard. It consists in introducing the requisite quantity of nitrate of soda directly into the chamber system, as, for instance, into the acid on the chamber floors or into the chamber atmospheres; similarly into the "absorbing tower" or "denitrating tower," &c. It may be admitted in its commercial state, in a dry powdery condition or in solution. If the chamber acid be passed through denitrating towers, then the nitrate of soda may be introduced on the floor of the chambers, preferably on that of the first in the series, by any suitable means, but the patentee prefers an aqueous solution, and that the flow or drip shall be regulated so as to be constant and not intermittent. It may also be injected into the atmosphere of the chamber by steam or other agency.

But if it be inconvenient to pass all the chamber acid made through a Glover's tower, then the above described method must not be followed, but the solution must be run into the absorbing or denitrating tower.

In whichever way applied, it is said that the nitrate of soda, even if it be not perfectly decomposed before reaching the denitrating tower, will there encounter quick and perfect decomposition, and in either case it will be decomposed and will readily yield its nitrogen acids and oxides.

By this practice the sulphate of soda is lost for the time in the process, but the patentee considers that a small matter, it being of little commercial value and mostly recovered in subsequent operations in which the acid is employed.

If it be intended to force the salt solution into the atmosphere of the chambers, this may be accomplished by a Gifford's injector or other apparatus, which might be so arranged that the liquid should be driven in as small spray.

Thus nitre-pots and ovens are done away with, and the nitrogen compounds are never allowed to come into contact with the sulphurous gases in the hot state in which they issue from the kilns.

Objections.—It has been objected to this invention that the sulphate of soda formed in the acid of the towers crystallizes out in the pipes and cisterns, if allowed to cool, and chokes them up, and even if it should not be in so great proportion as to do that, the acid thus impregnated cannot be used in the Gay-Lussac tower as it would soon fill the pores of the coke. This difficulty might be overcome by using only one Glover tower for this, and another for the nitro-sulphuric acid, but that would be impossible in works which did not possess two such towers, and would in any case be an objectionable restriction upon the working.

Difficulties in its Application.—Referring to the proposal of injecting the nitrate solution in the form of spray into the first chamber, Mr. Rennoldson at the Tyne Alkali

Works found it very difficult to adjust the steam jet, so as to convert the liquid into perfect spray, and to ensure the nitre remaining long enough suspended in the atmosphere of the chamber, to be completely decomposed before falling to the bottom. In his experience also so much steam was necessary, that, taken together with the steam evaporated from the Glover tower and the water from the nitre solution, it rendered the chamber acid far too weak.

To these objections the inventor has rejoined that he does adjust the steam with ease and accuracy, and the strengths of the chamber acid have, during the several years that the plan has been in operation, averaged about as follows:—No. 1, 130°; No. 2, 130°; No. 3, 120°; No. 4, 100°; Twaddell. It is also said the consumption of nitrate of soda has been by this process reduced to 1 per cent. on pyrites.

We have already alluded to Dr. Sprengel's spray system, which may also be adapted to the introduction of solutions or acids containing nitrogen into the chambers. The spray or mist may even be formed by the contact of two liquids at high pressure. On account of the small carrying power of the spray, Dr. Affleck says that the jets must be frequent, along the sides of the chambers for example.

Nitric Acid instead of Nitrate Soda.—Whereas in England nitrate of soda is practically speaking the only source of nitrogen employed in sulphuric acid manufacture, in France and Germany, on the other hand, as well as in most other countries probably, nitric acid is the universally adopted agent.

Dr. Lunge says the nitric acid is always introduced in a special small chamber where a terrace is built of earthenware dishes one above the other in the form of a staircase, so that the nitric acid, running into the topmost, flows over from a lip, runs into the second, overflows from it into the third, and so on until it reaches the bottom; and at the same time, the chamber gas, acting upon it, partly evaporates the liquid nitric acid, and partly changes it.

even then, into the well-known mixture of hyponitrous acid and nitric oxide, so that, when the acid arrives at the bottom of the terrace, it is so far diluted that it acts no more injuriously upon the chamber lead. The nitric acid is always introduced by means of vessels which allow a perfectly constant flow of it, similar to what was formerly done in the case of sulphuric acid flowing into the Gay-Lussac towers.

Further, he remarks (and his remarks are worthy of deep consideration as the result of very extended practical experience), that he is so convinced of the advantages of the system over the English method, that nothing but a fear of having too much difficulty with the workmen in the first instance would deter him from supplanting the latter system in England. The cost of fuel and labour in making the acid is exceedingly slight, and the great advantage of its use consists in the ease with which its supply can be made perfectly regular at all hours, and that it is always under control. The cost for nitre he found to be less in works (conducted with about the same degree of skill as ours), which used nitric acid, than in those using nitrate of soda as in England. The consumption of nitrate of soda (in the form of nitric acid) is often as low as $1\frac{1}{4}$ — $1\frac{1}{2}$ per cent. of the sulphur contained in the pyrites, and some German works profess to have got so low as 1 per cent.

Mr. Glover considers that the percentage of nitrate soda consumed bears an intimate relation to the cubic capacity of the chamber per ton of sulphur burnt, and that if the continental chambers were worked at a low figure, say 2 or 3 lbs. sulphur per 24 hours per cubic foot of chamber space, that would have much to do with the small consumption of nitre. But Dr. Lunge thinks that the chamber capacity in Germany is rather less than in England, which latter he estimates at about 4 lbs. per foot; and he feels convinced that when a certain limit has been passed, advantage will be gained neither in increased production of acid nor in de-

creased consumption of nitre, by any increment to the chamber space. This limit he thinks may certainly be put at 4 lbs. per cubic foot.

Of course denitrating apparatus is in use in the continental works whose nitrate of soda consumption is so low.

Notes on French Works.—Mr. R. Calvert Clapham has stated a few technical details concerning French manufacturing of his acquaintance. The pyrites used are generally non-cupreous, and contain 46—48 per cent. of sulphur. The charge in the kilns is light, not exceeding 7 cwt. per 24 hours; the kilns are carefully attended to, and leave an ore containing not more than $1\frac{1}{2}$ — $2\frac{1}{2}$ per cent. of sulphur. The yield of sulphuric acid, as might be expected under these circumstances, is very good, being about 135 per cent. on the pyrites. By this we suppose he means about 300 per 100 sulphur converted.

Cleaning Chambers.—Before proceeding further we will offer a few suggestions concerning the cleaning out of chamber floors. First of all the kilns must be suspended and when working with pyrites about 3 days should be allowed for the burning mass to die out after the last charge has been admitted. So soon as the chambers appear pretty free from gas, which may be seen by the windows and bell-glasses, the flues should be opened to their fullest extent, all lutes, bell-glasses, and windows removed, and man-holes should be cut in the sides or ends of the chambers of convenient size between the uprights. Thus an abundance of air is admitted to the chamber interiors. At the same time the acid on their floors should be drawn off to the last possible drop.

Underneath the acid will be found a layer of slimy-looking matter varying in depth according to the duration and conduct of the working, and consisting principally of sulphate of lead derived from the chamber lining and also of arsenic, tellurium, thallium, &c., in greater or less proportions

according to the class of pyrites used and the precautions taken to prevent the access of those substances into the chamber system. This chamber deposit will also contain appreciable quantities of nitrous and nitric acids which should be eliminated as far as possible before allowing workmen to enter the chambers, for asphyxiation may easily result from stirring up and breathing these poisonous gases. Danger of this kind may be avoided in a great measure by stirring the deposit thoroughly with long-handled wooden rakes so that the air may carry off the liberated fumes. When this has been pretty well effected, the workmen, shod in wooden sabots and provided with short wooden rakes and lights, if necessary, may enter and proceed to rake the muddy residue to a convenient spot for removal. The most convenient spot is generally the lowest point of the chamber floor, to which the semi-fluid mass will partly gravitate, and we found the most handy instrument for removing it to be an ordinary wooden corn-shovel. The men should be worked in short shifts, and the moment a man commences to cough, or feels a huskiness in his throat, he should be taken out into the fresh air.

When the cleaning is complete, and all necessary repairs have been made, the man-holes may be "burnt" up, windows, lutes, &c., replaced and secured, the needful quantity of weak acid run on to the chamber floors and the kilns relighted and charged.

CHAPTER III.

CHAMBER CONSTRUCTION.

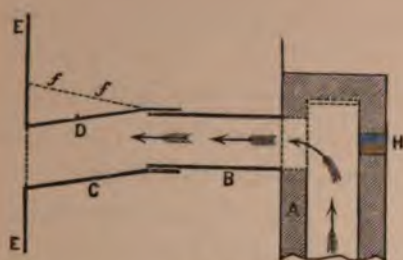


FIG. 18.

Connexion between Kilns and Chambers.—

When brimstone is used the gases from the kilns may be taken directly from the perpendicular kiln-stalk into the chambers, as shown in

fig. 18.

A is the kiln-stalk, *B* an iron pipe, *C* a leaden pipe, *E* side of chamber; arrows mark the course of the gas; *H* is a man-hole in the kiln-stalk, through which a flue-rake can be introduced, and the dust, &c., which accumulates in the pipe, *B*, can be raked to the bottom of the stalk, and from thence be removed. This can of course only be done when the kilns are not alight.

The leaden pipe, *C D*, which is shown in the plan, fig. 17, as *NO*, is not quite level, but has a slight inclination towards the chamber, so that any acid which may condense should not run back towards the kiln-stalk. If the sides of this pipe be parallel, the hot gases from the kilns will strike directly on the top at *D*, where a hole will soon be worn. This defect is best remedied by making the pipe rather funnel-shaped, with its largest opening towards the chamber; in this way, whilst the bottom of the pipe slopes towards the chamber as before, the top slopes

upwards, as shown by the dotted line *f, f*. The inclinations are somewhat exaggerated in the figure.

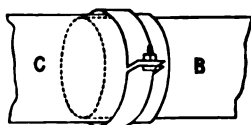


FIG. 19.

Fig. 19 gives an enlarged view of the junction of the pipes *B* and *C*. *N* is the cast-iron pipe (*B*), 22 in. internal diameter, connected with *O* (*C*), a leaden pipe 25 in. internal diameter,

secured where they lap by an iron band.

When using pyrites the gases are so hot that they must not be admitted immediately to the chambers, and they are best carried for some distance through iron pipes well ventilated as shown in fig. 13.

Cements.—Quick-lime and linseed oil mixed stiffly together form one of the best and hardest cements to put round the iron pipe leading into the chamber, under the lead that is secured on it by the iron collar. A stiff mixture of fire-clay and coal tar may also be used for the same purpose, as well as for the joints of stoneware pipes through which acid gases have to pass.

A cement which, according to Dr. Wagner, is proof against even boiling acids, may be made by a composition of caoutchouc, tallow, lime, and red lead. The caoutchouc must first be melted by a gentle heat, and then 6—8 per cent. by weight of tallow added, the mixture being kept well stirred. Next dry slaked lime is applied until the fluid mass assumes a consistency similar to that of soft paste, and lastly 20 per cent. of red lead is added, in order to make it harden and dry.

A solution of caoutchouc in twice its weight of raw linseed oil, heated and mixed with an equal weight of pipe-clay, yields a plastic mass resisting most acids.

Support of Chambers.—The construction of all leaden chambers is much the same in principle. They must be supported at a convenient height above the ground to admit of occasional inspection of their under surfaces; the framework is always of timber, and the

leaden sheets are joined together in the same way in all cases.

Mr. McCulloch adopts a plan of holding up the side lead which is ingenious and not commonly practised. Instead of the ordinary side rails between the chamber uprights to which the lead sheets are fastened by small square straps at intervals, he dispenses altogether with the side rails, and on each side of the chamber upright he burns a vertical strap on to the side lead of the chamber and of such a width that each pair of straps can be bent round the upright and lapped. This plan effects a great saving of timber and of labour, and the only objection which could be made to it, the risk of the lead tearing away at the crown-tree of the chamber, has never been found by Mr. McCulloch to have any practical existence. In working say four or five chambers in a set, he recommends the first two to be lined with 7 lb. lead and the others with 6 lb.

We shall now proceed to a minute description of the building and fitting of the four chambers shown in fig. 17.¹ We selected timber posts for supporting our chambers, they being cheaper than and preferable to either brick walls, brick pillars, or iron pillars: preferable because they occupy less space, can be strutted in case of need, and are more easily replaced.

The Timber Frame-work.—*Chambers A and B.* The timber posts underneath, measure 9 in. square; the centre upright is an iron column, tapering, diameter in the middle 6 in.; the 3 horizontal beams are 9 in. square; the floor joists 9 in. by 2½ in., and placed 12 in. apart, centre to centre; the four stouter joists, on which stand 20 of the uprights of the two chambers, are 9 in. by 6 in.; the floor boards are 1¼ in. thick; the 32 chamber uprights are 6 in. square; the horizontal rails between the

¹ In cold climates it is necessary to cover also the roofs of the chambers, and in that case there is no need for making the leaden roofs inclined, they may be horizontal.

uprights are, long rails 4 in. by $2\frac{1}{2}$ in., short rails $3\frac{1}{2}$ in. by $2\frac{1}{2}$ in.; the top plate or crown-tree is 6 in. square; the top joists are 9 in. by 3 in.

Chamber C has 21 timber posts underneath, 9 in. square; 7 beams 9 in. square; floor joists 9 in. by $2\frac{1}{2}$ in., about 12 in. apart centre to centre; 2 stouter joists which support the chamber uprights 9 in. by $5\frac{1}{2}$ in.; floor boards $1\frac{1}{4}$ in.; 44 chamber uprights 6 in. square; horizontal rails, short ones 4 in. by $2\frac{1}{2}$ in., long ones 4 in. by 3 in.; crown-tree 6 in. square; top joists 11 in. by 3 in. The floor is not quite level, having an inclination towards that corner of the chamber which is nearest to the syphon *j* in fig. 17, this being the lowest point by about 1 in. The greatest average depth of acid that this chamber is intended to hold is about $11\frac{1}{4}$ in. This quantity at its proper chamber strength of 113° Twaddell would weigh about 67 tons, or say 92 lbs. per square foot all over the chamber floor.

Chamber D has 9 timber posts underneath, 8 in. square; 3 beams 8 in. square; floor joists 9 in. by $2\frac{1}{2}$ in., about 14 in. apart centre to centre; 2 stouter joists to support 10 of the chamber uprights 9 in. by 6 in.; floor boards $1\frac{1}{4}$ in. thick; 28 uprights 6 in. square; horizontal rails 4 in. square; crown-tree 6 in. square; top joists 11 in. by 3 in.

Outside the posts supporting the chambers are smaller posts which carry the weather-boarding that surrounds the chambers, and encloses them from the weather (see plans, fig. 17), measuring 6 in. square. As these posts and those under the chambers rest on the ground, they have each a foundation block of concrete about 2 ft. square, and $1\frac{1}{2}$ ft. deep. Each post is steadied in its place on the concrete by



a pin (a piece of iron pipe) let into the bottom, and projecting down on to the concrete thus, and the top of each post has a tenon which is let into a mortice in the beam resting on it.

The chamber uprights are all morticed into the joists and beams on which they rest at bottom, and into the crown-tree at top.

The rails which support the side lead of the chambers by the leaden straps nailed to them are morticed into the chamber uprights in such a way that they can be removed if desired, the leaden straps being first unnailed and bent back thus :—

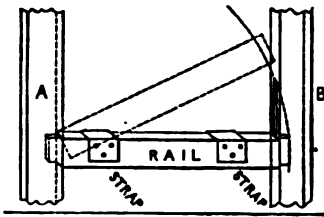


FIG. 20.

The long mortice in upright *B* allows this end of the rail to be raised until it frees the upright *B*, when the other end of the rail can be withdrawn from the upright *A*.

Those top joists (2 on each chamber) which are nearest to the crown-trees, and run parallel with them, are placed about 21 inches away from them. This we found to be a fault as the figures 21 and 22 will indicate. The side lead

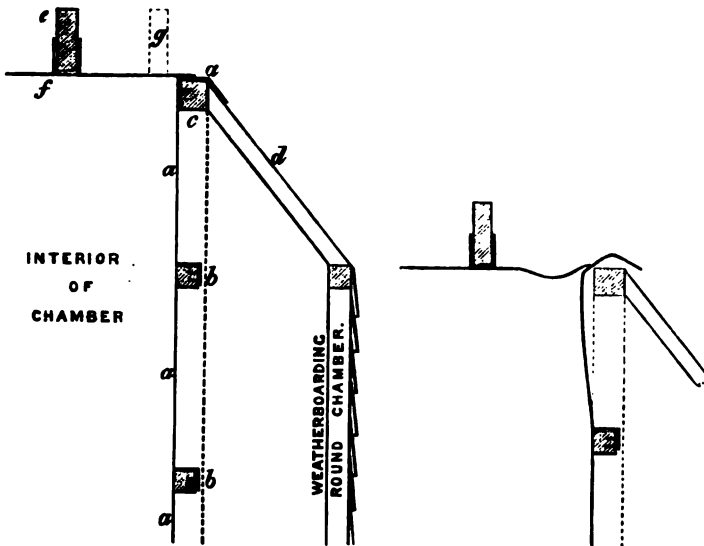


FIG. 21.

FIG. 22.

a (fig. 21) is supported by straps of lead fastened to it, and nailed on the rails *b*. The top end of the side lead is bent over the crown-tree *c*, and laps over the board-and-felt roof

d, but is not nailed, as nailing into the top of the crown-tree would admit wet, and rot the timber. The joist *e* (to which the top lead *f* is held by straps) being so far from the crown-tree *c*, the top lead by its own weight gradually sinks between the joist and crown-tree, and the side lead which is between the top rail and the crown-tree, having no support but its own stiffness, sinks with it, as shown in fig. 22. This would be prevented by placing the joist *e* at the spot marked *g*.

Lead Required.—The quantity of lead required for these chambers is as follows :—

CHAMBERS A AND B.		Square Feet. Inches.
Bottom : 15 ft. × 15 ft. + 3 in. to turn up all round = 15 ft. 6 in. × 15 ft. 6 in.	=	240 3
Top : 15 ft. × 15 ft. + 3 in. to lap all round = 15 ft. 6 in. × 15 ft. 6 in.	=	240 3
Sides : 15 ft. × 4 = 60 ft. × 13 ft. 6 in. average height + 9 in. to turn over at top all round = 60 ft. × 14 ft. 3 in.	=	855 0
		1335 6
Strips used for "burning," and loss at seam laps, say 3 per cent on 1335 ft.	=	40 0
Straps for top lead ; 9 joists × 14 straps to each = 126 straps, each 4 in. wide × 5 in. long + 1 in. for loss in burning = 6 in.	=	21 0
Straps for side lead ; 8 rails × 2 sides = 16 rails × 3 straps to each = 48 straps + 16 rails × 2 sides = 32 rails × 2 straps each = 64 straps = 112 straps × 4 in. × 6 in. as before	=	18 8
Total for one chamber	=	1415 2
		2
Total lead for Chambers A and B	=	2830 4

CHAMBER C.

	Square Feet. Inches.
Bottom : 60 ft. $\times$ 27 ft. 6 in. + 1 ft. to turn up all round = 62 ft. $\times$ 29 ft. 6 in. =	1829 0
Top: 60 ft. $\times$ 27 ft. 6 in. + 3 in. lap all round = 60 ft. 6 in. $\times$ 28 ft. =	1694 0
Sides: 60 ft. $\times$ 2 = 120 ft. + 27 ft. 6 in. $\times$ 2 = 55 ft. = 175 ft. $\times$ average height 23 ft. 6 in. + 9 in. for turning over at top = 24 ft. 3 in. =	4243 9
	7766 9
Allowance for strips and loss as above, say 3 per cent.	233 0
Straps for top lead : 33 joists $\times$ 27 straps = 891 straps $\times$ 6 in. $\times$ 4 in. =	148 6
Straps for side lead : 104 rails $\times$ 2 sides = 208 rails $\times$ 2 straps each = 416 straps + 28 rails $\times$ 2 sides = 56 rails $\times$ 3 straps = 168 straps = 584 straps $\times$ 6 in. $\times$ 4 in. =	97 4
	8245 7

CHAMBER D.

Bottom: 30 ft. $\times$ 23 ft. 6 in. + 3 in. to turn up all round = 30 ft. 6 in. $\times$ 24 ft. =	732 0
Top : ditto ditto ditto =	732 0
Sides: 30 ft. $\times$ 2 23 ft. 6 in. $\times$ 2 = 107 ft. $\times$ 22 ft. average height + 9 in. to turn over = 22 ft. 9 in. =	2434 3
	3898 3
Allowance for strips and loss as before, say 3 per cent =	117 0
Straps for top lead : 17 joists $\times$ 23 straps = 391 straps $\times$ 6 in. $\times$ 4 in. =	65 6
Straps for side lead : 24 rails $\times$ 2 sides = 48 rails $\times$ 3 straps = 144 straps + 48 rails $\times$ 2 sides = 96 rails $\times$ 2 straps each = 192 straps = 336 straps $\times$ 6 in. $\times$ 4 in. =	56 0
	4136 9

The total number of square feet of lead used is thus,
15,212 ft. 8 in., which at 6 lbs. per square foot = 40t. 14c.
3q. 24 lbs.

Joining the Lead Sheets.—The sheets of lead cannot be got beyond a certain size, in general they run about 7 ft. to 8 ft. wide $\times$ 30 ft. to 35 ft. long as a maximum figure. In consequence of this some artificial manner of rendering them one entire piece must be adopted. The method now in universal practice is that of fusing the sheets together by the aid of the apparatus described below. This apparatus consists of a hydrogen gas generator, or burning machine, as it is vulgarly called, an air vessel or

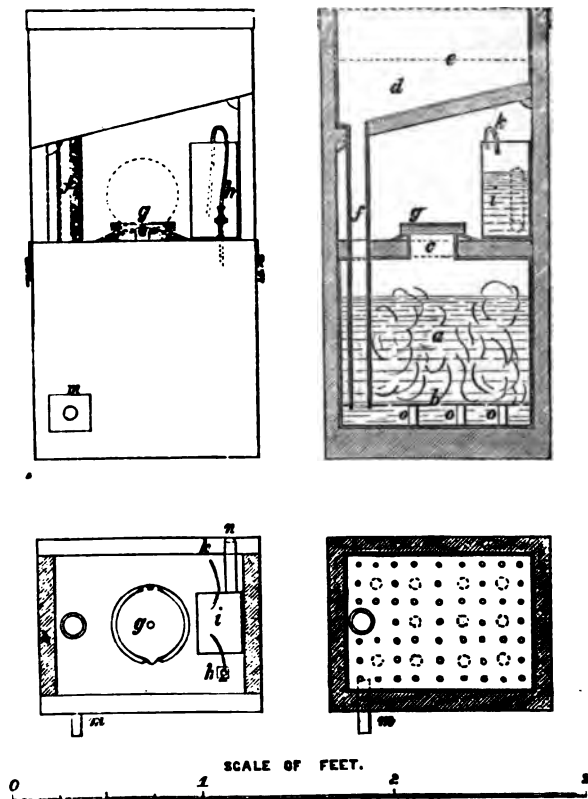


FIG. 23.

portable bellows, some india-rubber tubing and a set of gas cocks and jets. The hydrogen generator is shown in fig. 23.

a is an air-tight leaden cistern, having a perforated shelf,

b, and an opening in the top, *c*; *d* is another leaden cistern with a perforated shelf, *e*. A pipe, *f*, connects the cisterns, *a* and *d* passing through *a* as far as the shelf *b*, which it just perforates.

The hinged cover, *g*, being turned back, the cistern, *a*, is filled with sheet zinc cuttings, and the cover closed. Diluted oil of vitriol (say O. V. 1 quart to water 1 gallon) is poured into the cistern *d*, and finds its way through the pipe *f*, into the bottom of the cistern *a*, rises through the strainer *b*, and surrounds the zinc. As the hydrogen gas is formed, it passes through the cock, and pipe *h* into the leaden vessel *i*, partly filled with water, and passing through the water it becomes purified, and escapes at the pipe *k*. *m* is the pipe through which the generator is emptied of the acid when gas is no longer required. The vessel *i* may be removed from its place by unscrewing the nut close to the cock on the pipe *h*, and may be filled with water or emptied through the pipe *n*. The pipes *m* and *n* are plugged with corks. *o* are short pieces of pipe supporting the shelf *b*, to which they are attached.

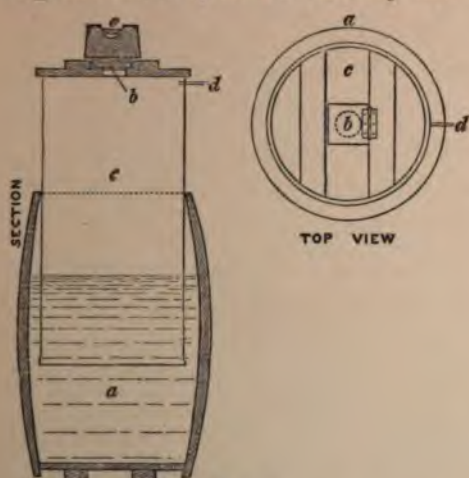


FIG. 24.

The air vessel consists simply of a wooden cask open at the top containing a cylinder of zinc, with a closed top, having a hole and cover in the centre, see fig. 24, drawn to half inch scale. The cask *a* is partly filled with water, the cover *b* (which is coated un-

derneath with sheet india-rubber to make it shut close) is opened, the cylinder *c* raised and the cover closed again, preventing the escape of air from the cylinder except

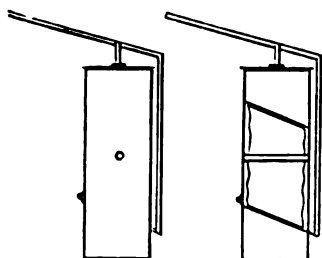


FIG. 25.

through the small pipe *d*. A weight, *e*, is placed on the top of the cylinder which keeps the cover *b* firmly closed and gives force to the current of air issuing from *d*, the weight being 28, 56, or 112 lbs., according to the pressure of air required.

Fig. 25 shows a portable bellows sometimes used by plumbers for obtaining a supply of air. It is more portable than the air vessel, but cannot be used without an assistant to work it.



FIG. 26.

India-rubber tubes (*a*, *b*, fig. 26) connect the gas generator and air vessel or bellows with a pair of brass cocks, and breeches pipe *c*. The gas and air being admitted through these cocks, unite in the tube *d*, and passing through the brass pipe and jet *e*, *f*, may be ignited and produce an intensely hot flame, by which the lead sheets may be joined without the aid of any flux. The lead to be "burnt" must first be scraped bright, and where a strong seam is required, as for instance on the bottoms of chambers, strips of clean lead are run on in the manner of solder. Several jets are in use, with holes of various sizes for procuring a large or small flame, according to the special requirements of the work in hand, and the intensity of the heat is also regulated by the proportions and quantities of gas and air admitted through the cocks. As it is essential that the flame should

not be subject to sudden variation, little brass tubes are fitted to the nozzle, to guard the flame from air currents when working out of doors, or in draughty places.

Raising the Lead Sheets.—After the wooden framework of the chambers is erected, the following apparatus will be required for getting the sheets of lead into place, viz. a hoisting board, a pair of blocks and fall, a snatch block, a crab winch, trestles and scaffold boards, a scaffold pole and 2 ladders (long and short).

The hoisting board, fig. 27 is made as long as the sides

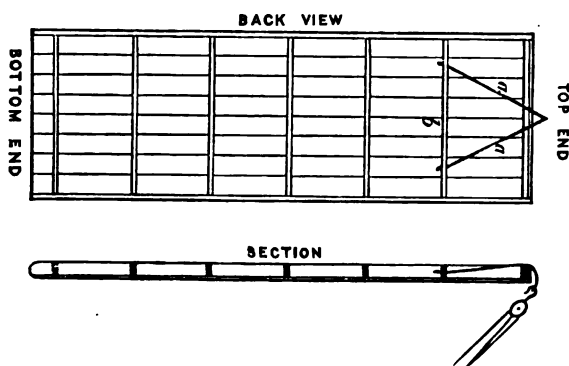


FIG. 27.

of the chamber are high, and as wide as the width of one or two sheets of lead, according to whether it is intended to raise one or two sheets at a time. If two sheets are hoisted at once, their edges are joined (making them one) before hoisting. The figure represents the back view and section of a board 21 ft. long by 7 ft. wide. The cross pieces are morticed into the side pieces, but not nailed, so as to be more easily taken apart for removal and reconstruction in the various chambers in succession. The framing measures 6 in. 2 in., on which is lightly nailed 1 in. boarding. *a* is a rope to the middle of which, at the top of the board, the hoisting tackle is attached. The ends of the rope pass through the cross piece *b*, and are secured by knots.

Side Lead.—The side lead is the first put up in a chamber,

then the top lead, and lastly the bottom or floor lead. Commencing with the side lead, the hoisting board is laid flat on the floor of the chamber with its face upwards. A sheet of lead is then unrolled on the board so that one end of the sheet is even with the bottom end of the board, whilst the other end extends about a foot beyond the top end of the board. The straps of lead by which the sheet is to be held to the framework of the chamber, are fused on to the sheet, their proper situations having been first determined, and marked with a chalk line. The straps may be about 4 in. square, and of the same stoutness as the sheet. They are so placed that when the sheet is reared into its position they can be bent down and nailed to each cross rail of the chamber against which the sheet hangs (see fig. 20, showing only one cross rail). About 18 in. of the bottom end of the sheet is now folded back (see fig. 28) so that

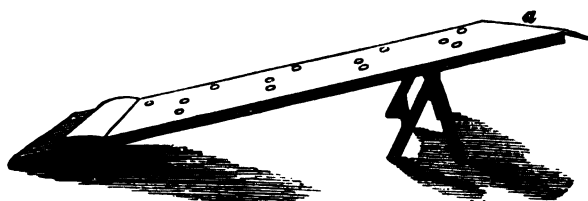


FIG. 28.

when the sheet is up it may not reach the floor by that amount. This is done for greater convenience in afterwards laying the bottom or floor lead of the chamber, which is turned up about 12 in. or 15 in. all round the

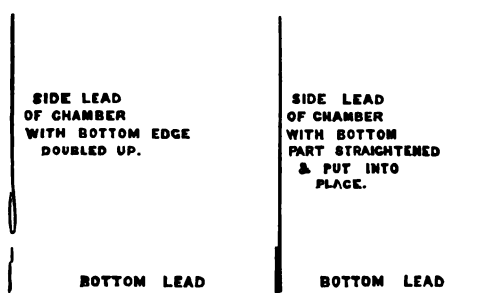


FIG. 29.

chamber (forming a shallow cistern, as it were) the side lead being afterwards unfolded to hang down inside it, see fig. 29. Fig. 28 shows the board partially

raised for the purpose of securing the sheet to it by turning it over the edge at *a*, and nailing it to the back of the top rail of the board. This being done, the rope at the back of the board is attached to the hoisting tackle by which it is reared into its place,—see fig. 30.

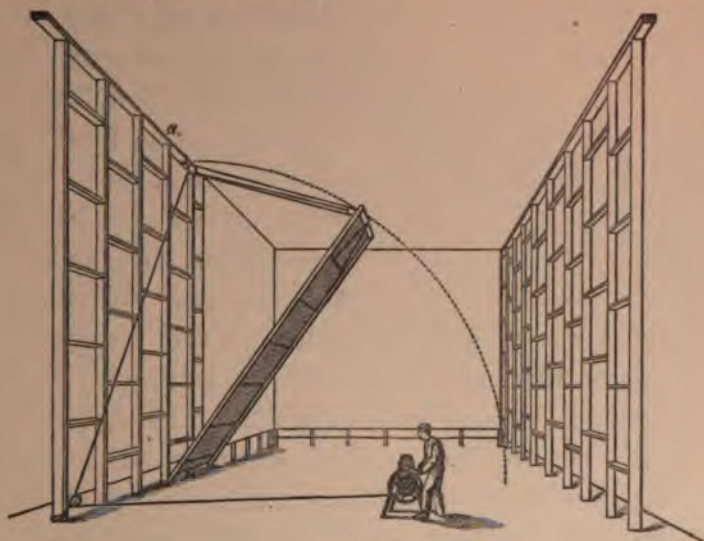


FIG. 30.

The straps are then all nailed to the chamber cross rails, and the top of the sheet is unfastened from the hoisting board and bent over the crown-tree or top plate of the chamber (*a*, fig. 30), but not nailed, as the sheet is already sufficiently supported by the straps.

Each sheet overlaps about 2 in. the preceding sheet, and all the sheets are joined by fusing the overlapping edges to the lead which they cover.

Top Lead.—When the side lead has been erected all round a chamber, a temporary platform must be erected inside, and nearly as high as the top of the chamber, on which the top-lead may be rolled out and supported whilst being joined and fastened to the top joists of the chamber. This platform is made with trestles and boards. The trestles are formed of quartering 6 in. $\times$ 2 in., each one (see fig. 31) consisting of a bottom and top piece and three or

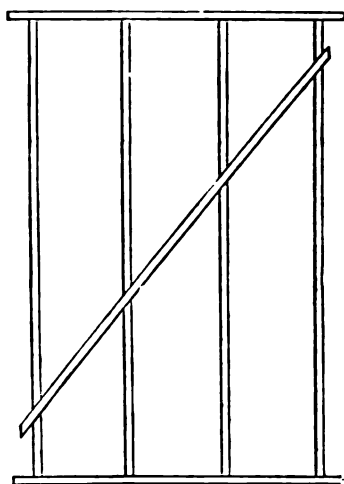


FIG. 31.

four uprights, the latter being morticed into the former, but not nailed. A diagonal brace is nailed to the uprights, to keep them in position.

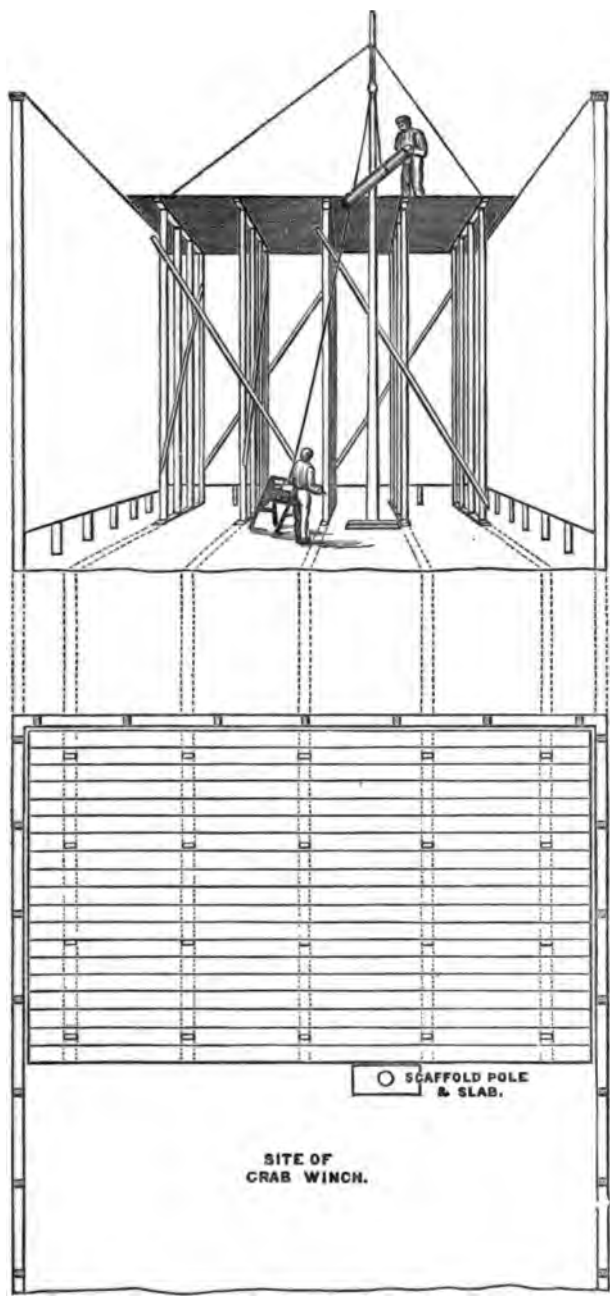
The trestles are stood upright about four feet apart across the chamber, and kept in position by diagonal bracing from one to the other. Boards are laid close together on the top of the trestles, and nailed where

absolutely necessary, and the platform is then complete.

The top of the platform should be level with the top of the chamber crown-tree (see fig. 32). This figure shows the platform erected at one end of a chamber, and a sheet of lead being raised to commence covering it in. Each trestle stands on three or four small blocks of wood about 4 in. deep. When it becomes necessary to move the platform forwards to a fresh place, these blocks are taken away and small rollers of wood, or short iron pipes about 2 in. in diameter are substituted. When this is done the platform stands 2 in. lower than before, leaving a clear space between it and the top lead, and can be easily moved forwards on the rollers. Figure 33 is a plan of the platform showing the top boards.

The leaden straps by which the top and side lead of chambers is supported should be sufficiently large and numerous to hold the lead securely, and as regards the top lead to keep it as level as possible and prevent its bagging down in hollows. On the top lead the straps should be not less than 4 in. and not more than 15 in. from centre to centre along the joists (see fig. 34).

On the side lead the straps may be the same width (say



Figs. 32, 33.

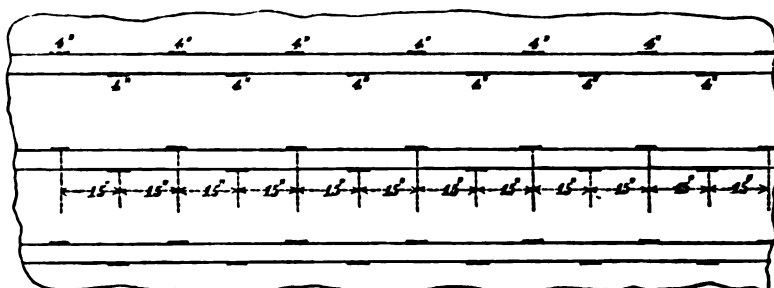


FIG. 34.

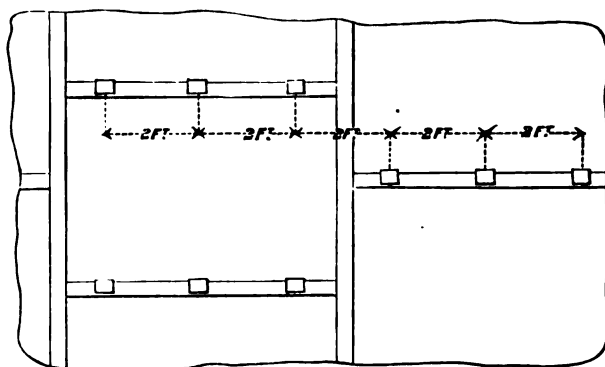


FIG. 35.

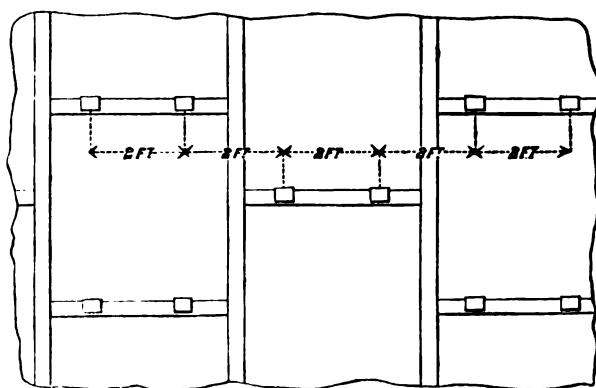


FIG. 36.

4 in.) and not more than 2 ft. apart from centre to centre (see figs. 35 and 36).

When the top of the chamber has been covered in, the platform is carefully taken down and removed. The bottom sheets of lead are then unrolled, their edges joined

to form the bottom of the chamber, and a piece turned up all round, 6 in. or 12 in. high, or more, according

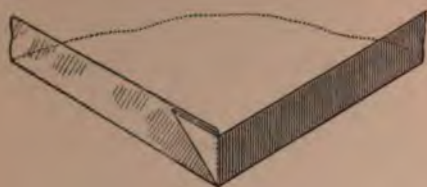


FIG. 37.

to the quantity of acid the chamber is intended to hold. At each corner of the chamber this turned-up lead is bent and folded so as to form a corner without

making a seam (see fig. 37).

When the bottom of the chamber is completed, the side sheets are straightened down into place (see fig. 29), and the upright seams on the side lead finished to the bottom of the sheets. The plumber (now completely enclosed) makes his exit through a hole cut in the side lead, which he afterwards closes by a patch on the outside.

To Stop a Leak.—The escape of gas must be stopped before the hole can be repaired. To do this take a piece of brown paper, cover it with pitch, and put it over the hole; this will stop the escape for from 12 to 24 hours, during which time a patch of lead may be “burnt” on to cover the hole.

Testing Lead.—It is important that the lead used for building chambers, &c., should be tested by an analyst to ascertain whether it is free from an appreciable amount of tin and antimony, say more than $\frac{3}{4}$ per cent. (0.75). These metals, if they are present to any large extent, being readily acted upon by sulphuric acid, set up galvanic action, and hence cause the lead to corrode and become eaten away, small holes being chiefly but continuously formed. Samples of the lead used by us gave by assay, tin 0.34 per cent., and antimony, traces.

Stoutness and Weight of Lead.

Milled sheet lead 7 lbs. per square ft. = about $\frac{1}{8}$ in. thick

“ “ “ 14 lbs. “ “ “ = “ $\frac{1}{4}$ in. “

“ “ “ 21 lbs. “ “ “ = “ $\frac{3}{8}$ in. “

Wherever lead is exposed to the action of the chamber gases, it should not be lighter than 6 lbs. per square foot. The corroding action of the gas is greatest in the connecting trunks and chamber condensers. In the chambers, the top lead wears out first, especially along on the crown-trees, then the side lead, and lastly the bottom lead, owing perhaps to its being protected by the layer of sulphate of lead which soon covers it. It is usual, however, to have the bottom lead stouter, if anything, than the side and top lead, as a leak in the bottom is productive of the greatest inconvenience and expense, which it is as well to guard against.

Lead pipe.—Patent hydraulic lead pipe may be got of the following bores, weights, and lengths :—

Weight per yard.					Weight per yard.				
Inside Dia.	Light.	Middle.	Stout.	Length.	Inside Dia.	Light.	Middle.	Stout.	Length.
$\frac{3}{8}$ in.	2½lbs.	3½lbs.	4½lbs.	50yds.	$1\frac{3}{4}$ in.	15lbs.	18lbs.	22lbs.	10yds.
$\frac{1}{2}$ "	3½ "	4½ "	5½ "	45 "	2 "	20 "	25 "	30 "	6 "
$\frac{5}{8}$ "	4½ "	5½ "	6½ "	36 "	2½ "	25 "	30 "	35 "	5 "
$\frac{3}{4}$ "	6 "	7½ "	9 "	24 "	2½ "	30 "	35 "	40 "	4 "
$\frac{7}{8}$ "	7 "	8½ "	10 "	22 "	3 "	32 "	42 "	52 "	10 ft.
1 "	8 "	10 "	12 "	20 "	3½ "	46 "	51 "	56 "	10 "
1¼ "	11 "	13 "	15 "	14 "	4 "	52 "	63 "	74 "	10 "
1½ "	13 "	15½ "	18 "	12 "	4½ "	60 "	70 "	80 "	10 "
1½ "	14 "	16½ "	19 "	10 "					

Plumbers' Charges.—Plumbers who can erect chambers by the autogenous process (as hereinbefore described) will do all the principal work at a price varying from about 20s. to 30s. per ton of lead used; but the small work, such as fittings, &c., at per day.

Chamber Fittings—Top Lights.—Fig. 38 is a vertical section of a glass bell jar (common propagating glasses



FIG. 38.

The jar, *a*, is luted to the top lead, *b*, by the water contained in the channel formed by the turned-up edge of the lead, *c*, and the leaden collar, *d*, which is fused on to the top lead.

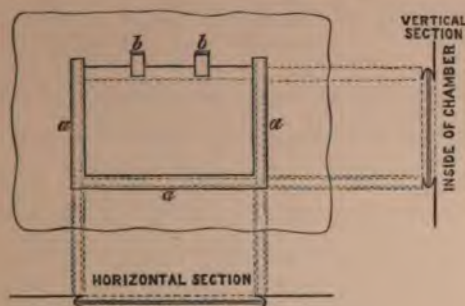


FIG. 39.

rather smaller than the piece of glass, and a strip of lead folded, cut into three pieces, and joined at right angles, forming the frame *a*, into which the glass will slide. The back edge of this frame is "burnt" to the bottom and side edges of the hole in the chamber. The glass is then put in and the clips of lead, *b*, are fused to the chamber lead and bent over the glass, so as to keep that part of the lead and glass in close contact. The window is then made gas-

answer the purpose admirably), such as are placed on the top of a chamber over a hole in the top lead, to admit light and thus render the colour of the gases visible.

Windows. —

Fig. 39 is a chamber window, or pane of glass fixed, in the side lead of a chamber, through which to observe the colour of the gases inside. A hole is first cut in the lead

tight by stopping it all round with white and red lead.

Drip Trays.—

Drip trays are placed in chambers to catch the condensed gas and indicate the rapidity of the condensation and the strength of the acid condensed, see fig. 40. *a* is the tray of stout lead, sup-

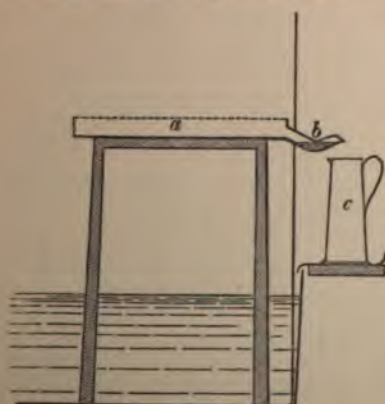


FIG. 40.

ported at a convenient height above the floor of the chamber. The acid which collects on the tray is conveyed by the small pipe, *b*, through the side lead of the chamber, and drops into the leaden cup, *c*, which stands on a tray a little higher than the turned-up bottom lead of the chamber, so that when the cup overflows the acid runs back into the chamber. The pipe, *b*, is bent, so that a little acid always remains in it and prevents the escape of gas from the chamber.

Gauge Glass.—The gauge glass or float is a small glass tube or stem enclosing a scale of inches (subdivided into tenths) and terminating in a spherical glass bulb or float. It is used for ascertaining the depth of acid in a chamber at any time more conveniently and exactly than could be

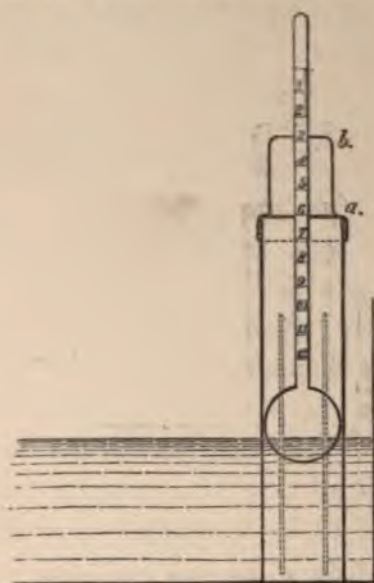


FIG. 41.

done by dipping a rule into the acid. Fig. 41 represents a vertical section of a gauge glass placed in position between the side lead of the chamber (which has been pressed inwards to obtain sufficient room) and the up-turned bottom lead. The glass is enclosed in a leaden cylinder with perforated slits in its sides to admit the entrance of the acid on which the bulb floats. The cylinder has a movable cap or cover, *a*, on which is fastened a

guide or strip of lead, *b*, each having a hole in its centre through which passes the stem of the glass. This arrangement secures the perpendicular position of the glass, and allows it to move freely up or down with the rise or fall of the acid. The reading of the scale of inches at the level of the top of the cap, *a*, corresponds with the depth of acid in the chamber. In the figure this appears to be 6 in.

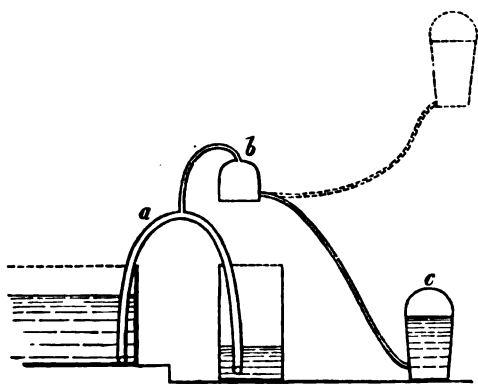


FIG. 42.

Syphon Setter.—A syphon setting apparatus is shown in fig. 42; *a* is the syphon, *b* a closed leaden vessel, *c* an open vessel or bucket. A small metal pipe connects the top of the syphon with the top of the vessel, *b*, and an india-

rubber tube connects the bottom of *b* with the bottom of *c*. To set the syphon (both legs of which must be standing in liquid), fill the bucket with water and raise it above *b*, and hold it there till all the water has run into the vessel *b*. Then stand the bucket down, and the water will flow back into it, and the vacuum thereby created in *b* will exhaust the air from the syphon and set it running.

Uniform Flow under Varying Pressure.—To obtain an uniform flow of acid or other liquid under varying degrees

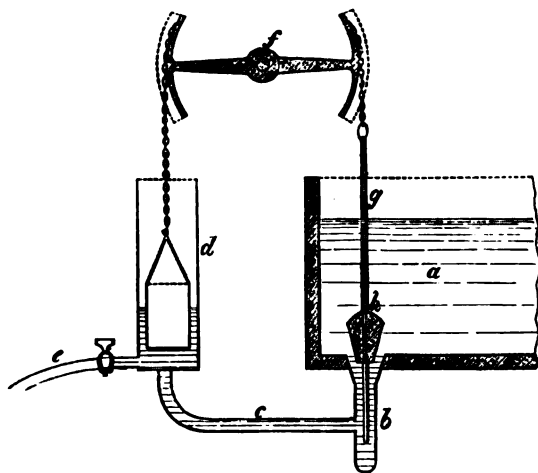


FIG. 43.

of pressure the apparatus shown in fig. 43 is used. *a* is a cistern containing acid, *b* a well in the cistern having a

conical mouth, *c* a pipe connecting the well and cistern with the cylindrical vessel *d*, which must be the same height as *a*, *e*, a delivery pipe fitted with a tap, *f*, a lever working on a central pivot. One end of the lever is attached by a chain to a leaden bucket which hangs in the vessel *d*, and the other end is attached by a short chain to the rod *g* (which is of iron cased in lead), having cast on it the conical plug *h*, which is to fit accurately the contracting mouth of the well *b*. The extension of the rod below the plug serves to keep the latter in its seat. The tap in the pipe *e* being opened, the greater the pressure of acid in *a*, the higher it will rise in *d*, elevating the bucket and depressing the plug *h*, which will check the flow. Thus at whatever height and consequent pressure the liquid in the cistern may be, the pressure or flow of acid from the delivery pipe will be uniform.

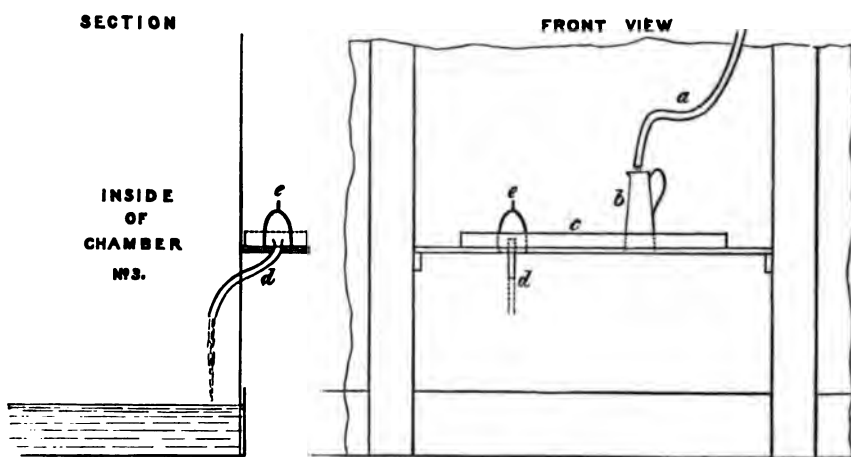


FIG. 44.

Sampling Chamber Acid.—Fig. 44 shows an arrangement for ascertaining the strength of the acid which flows from chambers *A* and *B* into chamber *C*. *a* is a pipe conducting the overflow of acid from *A* and *B*, *b* a cup to catch the acid for its strength to be tried, *c* a leaden tray on a wooden shelf against the side of the chamber *C*, from

whence it flows through the pipe *d* into the inside of chamber *C*. *e* is an inverted leaden cup with notched edge which lutes the pipe *d*, and prevents the escape of gas through it.

Putty.—An excellent putty for stopping round pipes, manhole doors, &c., in chambers consists of an admixture of china clay and boiled oil.

Plumbing Hints.—To ascertain the correct size to cut a piece of sheet lead in order to make a cylinder of any exact diameter required. Multiply the internal diameter required by 3.1416, and add twice the thickness of the lead. Example: Required a cylinder $2\frac{1}{4}$ in. internal diameter, made of sheet lead $\frac{1}{8}$ in. thick. Then $2.25 \times 3.1416 = 7.0686$ in., say 7 in. + $(\frac{1}{8} \times 2) = 7\frac{1}{4}$ in. In this case the edges of the lead are made just to meet where the seam is made, but in cylinders exceeding say 6 in. diameter, it may be preferable, unless the lead be very stout, to lap the edges one over the other about 1 in. or $1\frac{1}{2}$ in., which must be allowed extra in cutting out the lead.

To make a leaden funnel. Take a piece of lead as wide as the intended diameter of the mouth of the funnel, and twice as long, and in it strike a half-circle as large as the lead will allow. Cut the lead where so marked, and also cut out a small semicircular piece as at *a* (fig. 45). Bend the lead so that the edges *b* and *c* meet, and join them. A piece of lead pipe joined to the apex of the cone will complete the funnel.

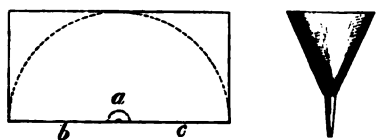


FIG. 45.

CHAPTER IV.

CONDENSERS AND COLUMNS.

It will be remembered that the nitric acid gas does not enter into the composition of the sulphuric acid, but that it is simply an agent or go-between, effecting or assisting in the reaction of the other vapours upon each other, and that, theoretically, a given portion once admitted to the chamber should last for ever, and still remain nitric acid gas. In point of fact it does retain its nature to the end, and a great portion of it goes away at the exit from the chambers in precisely the same state as when it entered. Nitre being one of the most costly items in the manufacture of sulphuric acid, many methods for reducing its proportions have been introduced from time to time, and it is claimed for some of them that they bring down the percentage required of nitre to sulphur to about 3—2, and even 1 per cent.

Substances Absorbing Nitrous Gas.—Various substances are used for catching (by absorption) these escaping nitrous fumes. Milk of lime is sometimes employed, and the nitrous gases are brought into contact with it, and form nitrate of lime. In other cases nitrate of barium is produced by exposing witherite (natural carbonate of barium), finely ground and suspended in water, to the action of the gases. Even pure water is occasionally made to absorb the gases by being allowed to trickle down large high towers somewhat akin to coke condensers in construction.

Another plan, which may be adopted with considerable profit when the manufacturer chances to be near a gas-works, consists in allowing a stream of ammoniacal water

from gas-works, commonly called "gas liquor," to filter and trickle down a column filled with coke or flints, up which the waste gases from the chambers are made to ascend. The ammonia is converted into sulphate of ammonia, and, where there has been an excess of sulphurous acid, it is found that, on heating the liquor in open air, the sulphite and hyposulphite of ammonia rapidly pass into sulphate.

Sulphuric Acid as an Absorber.—But of all the substances applied to the purpose, sulphuric acid itself is, undoubtedly, the general favourite. This acid at a specific gravity of 1.711 will absorb about $2\frac{1}{2}$ per cent. of nitrous anhydride, while at 1.845 sp. gr. (the strongest concentrated oil of vitriol) it takes up nearly 8 per cent. Acid possesses this advantage, also, that it can be readily admitted to the chambers or other receiver, and there made to give up its nitrous gases, for direct re-use in the chambers.

In Stoneware Jars.—One method of bringing the gases and the sulphuric acid into contact consists of a series of stoneware jars. This plan is in favour principally on the continent, and is scarcely known in this country. The plates (fig. 16) show the general arrangement of the jars in relation to the chambers, and the manner of working them.

Fig. 46 shows an enlarged view of two jars and the necessary pipes, as manufactured by M. Stanislas Godin, Chapelle-aux-pots, près Beauvais, Oise, France.

A A' jars with two openings of 25 centimetres ($9\frac{3}{4}$ in.) inside diameter.

B B elbows of communication connecting the jars.

C small pipe descending to the bottom of each jar inside, serving to ascertain the level of the water or acid in the jar, and also to syphon the acid from the jars.

D D small openings of 8 centimetres (3.12 in.) diameter, serving to support the small syphon pipes *C*.

E E E E small tubes placed at 25 centimetres ($9\frac{3}{4}$ in.)

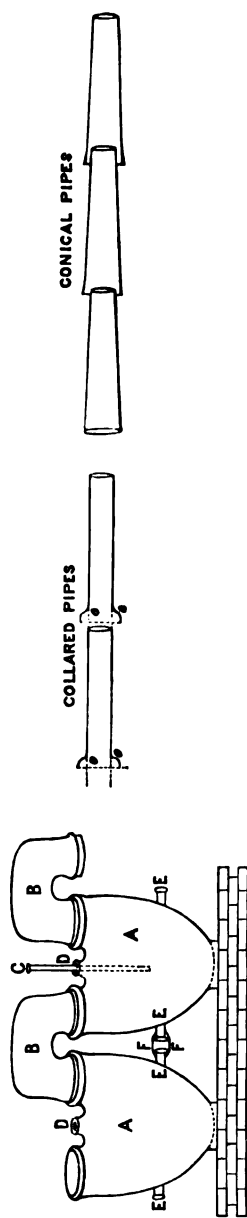


FIG. 46.

from the bottom of the jars, having 2 centimetres (0.78 in.) inside diameter, and connected by means of a small india-rubber tube, *F*. They serve to make the acid of one jar communicate with another, and by this means it is easy to draw the acid of the whole apparatus through one jar.

The cubic capacity of each jar is about 200 litres, or 44 gallons.

The collared pipes are 1 metre (39.37 in.) long and 20 centimetres (7.8 in.) diameter at the smaller end, and one is inserted in the other as far as the point shown by the letter *o*.

The conical pipes have the same length and diameter, and may be inserted as far as desired.

The price of each jar is 25fr. or 1*l.*, f.o. rail at Beauvais, or f.o. quay at Rouen. In that price are included three accessories, an elbow, *B*, a pipe, *C*, and a packing-basket. The price of each pipe, either straight-sided or conical, is 4fr. 25c. or 3*s.* 6½*d.*, packing included, either f.o.r. at Beauvais, or f.o. quay at Rouen.

A kind of putty would be required for luting the elbows to the jars. Mons. Godin would either send the recipe of its composition or obtain the putty.

To make 70 jars and their accessories, a period of about six weeks would be needed.

Recovering Nitre from Acid.—M. Payen recommends the acid to be put into these jars at a strength of 1.732 sp. gr.,

and only allows it to remain three days exposed to the gases, when it must be subjected to some process for extracting from it the nitrous gases which it is holding. It is questionable whether it would be good to introduce the nitre-saturated acid simply into the chambers. Merely diluting it with hot water, however, answers moderately well, and this may be done within the chamber. An apparatus that may be conveniently employed for this purpose consists of a round leaden vessel about 12 in. high and 18 in. in diameter. This is placed close to the leaden side of the chamber, which is pierced by three tubes, all terminating inside the leaden vessel and near its bottom. One of these tubes conveys the nitriferous sulphuric acid, a second brings water, and the third steam. The two tubes for the liquids terminate on the outside of the chamber in funnels, while above these funnels are the taps respectively of the nitriferous acid cistern and the water supply. The steam pipe naturally comes from a boiler.

Supposing the chambers to be at work, the mode of operating with this arrangement is very simple. The little leaden receptacle is first run full of water, and this is then brought to the boiling-point by turning steam into it. The workman then proceeds to admit slow streams of the nitriferous acid and water from their respective taps. Immediately this acid touches the hot water in the receptacle it decomposes into simple sulphuric acid and nitrous gases—the former mixing with the acid already in the chamber, and the latter ascending and combining with the kiln gases to assist in a further production of sulphuric acid.

Of course the vessel is always nearly full, the streams of fresh liquid rushing in immediately and constantly displacing a portion of the previous contents.

At least two advantages arise from having this decomposer as near the entrance of the sulphurous acid gas as possible. In the first place the gas arising from the decomposition has a better chance of thoroughly mixing with

the rest of the gases which form the chamber mixture; and in the second place, rapid removal and immediate mixture lessen the risk of local action on the chamber, nitrous gas being rather destructive to lead. In fact, the rapid current of sulphurous acid gas should sweep away every particle of nitrous gas as fast as it is liberated.

Muspratt says that after one of these decomposers had been working in a chamber for nearly three years, the lead in its immediate neighbourhood did not appear to have suffered more than that in any other part of the chamber.

Coke Condenser.—It is only in a large works that the apparatus invented by Gay-Lussac and Glover, which we shall presently describe, can be used to advantage, a small manufactory must be content with a set of Woulfe's jars, as already described, or with a common old-fashioned coke condenser, such as the following.

The shape may be either square or cylindrical: perhaps the former is better on account of more easily supporting the lead. The height may be about 20 ft., and the diameter 4 ft. 6 in. The column consists of a stout wooden framework well morticed together, and provided with cross-rails (as the chambers), for supporting the sheet lead sides. The lead should be at least 7 lbs. to the foot for the sides, and 9 lbs. per foot for the top and bottom; the sides should also be lined with fire-tiles to prevent scratching of the lead. In some cases also the space between the lead and the fire-tiles is stuffed with a mixture of fire-clay and tar. At the bottom should be placed fire-bricks, loosely arranged without cement of any kind, so as to leave a free way for the ascent of the gases, by preventing the coke from sagging down. Iron bars, coated with thick lead, are laid across the fire-bricks, and the coke rests immediately on them. The coke must be carefully picked as described on a subsequent page, and should be packed in at the same time with the fire-tiles,

as in a Gay-Lussac or other tower. The gas enters near the bottom of the tower and traverses the whole height of the column, that which remains of it escaping at the top may be taken direct to the chimney, or, what is much better, passed first *down* a second similar column. The acid drips to the bottom of the condenser and flows out through a pipe, whence it may be conducted to the working chamber, and there introduced in an atomized form by the application of Dr. Sprengel's spray producer. On this pipe should be fixed a perforated cup-shaped cap, to prevent the pipe becoming choked by the entrance into it of coke dust, &c. A circular hole may be made in the side lead, large enough to admit a man's hand, if required to remove any internal obstruction to the flow of the acid. This hole is closed by a cover resting in a collar, and the escape of gas is stopped with red and white lead, or other suitable luting material. The bottom lead of the condenser is turned up 5 or 6 in. all round. It is well to have a luted hole at the top of the tower.

One such tower will cost about 60—70*l*.

Gay-Lussac Columns.—The apparatus invented by Gay-Lussac, and named after him, is applied in its original or a modified form to the economization of the nitrous gases in almost every large establishment.

It consists essentially of two separate and distinct parts:—

1. A column placed between the escape pipe of the chambers and the chimney, for catching the nitrous gases in their exit, known as the “absorbing tower” or “absorber,” and

2. A column placed between the kilns and the entrance to the chambers in which the acid from the “absorber” is robbed of its nitrogen compounds, known as the “denitrator.”

Absorbing Tower.—The “absorber” may be built of stout timber lined with thick sheet lead protected inside

by a course of fire-tiles set in fire-clay and packed with coke of the hardest possible description broken into medium-sized pieces, and selected free from dust, or it may be filled with broken wine bottles or other convenient materials. The coke or other substance should rest on fire-bricks set on edge at a little distance apart.

The chamber gases enter at the bottom of the tower, while sulphuric acid, generally at a strength of about 150° Twaddell, is supplied at the top. The acid and the gases are thus brought into contact with each other in a divided state, and the acid will absorb more or less of the nitrous gases contained in the mixed vapour. The non-absorbed residuum of the gases escapes into the chimney, while the nitre-saturated acid flows from the bottom of the tower into a suitable cistern.

It has been generally calculated that 1 ton of sulphur converted per 24 hours would require a column about 30 ft. high and 2 ft. 10 in. diameter. A larger consumption of sulphur would require a larger tower, and as the absorption was most completely effected in a narrow and high column, it was thought preferable to increase the height rather than the diameter of the tower, which latter was never allowed to exceed 3 ft. on account of the difficulty experienced in distributing the acid uniformly over the whole of a large surface with the means then provided. When the consumption of sulphur was so great that sufficient elevation could not be conveniently given to the tower, recourse was had to two columns of small diameter rather than a single large one, and two escape pipes from the chambers.

Segner Wheel.—By using the Segner wheel, however, first introduced at Aussig, in Bohemia, some years since, the acid may be evenly distributed over any desired superficial area, whether confined or extended.

The top of the tower (fig. 47) is covered with a sheet lead dish (*a*) turned up about 4 in., high round the edges, and divided equally into 16 separate compartments by means of



FIG. 47.

16 little leaden partitions, *b*, burnt on to the dish. As will be seen by the figure the dimensions of these compartments, *c*, are calculated in such a way that each contains exactly the same quantity as the others, and at the same time their shape is such that those portions of the partitions which come within the range of the wheel are exactly equidistant from each other. Each of these compartments is furnished with a tubelet about $\frac{1}{2}$ in. high, *d*, covered with a leaden capsule which surrounds the tubelet and is provided with slits about $\frac{1}{3}$ in. high, through which the acid can pass unhindered while the gas is prevented from escaping. The acid is supplied from a cistern, *e*, through the pipe, *f*, furnished with a cock, *g*, and is admitted to the top of the Segner wheel, *h*. The lower part of the "wheel" and both arms, *i*, are of lead, above which is fixed a thick glass tube terminating at the summit in a funnel, while below the leaden

pipe is another short glass rod which is drawn to a point at the bottom, the whole revolving in a little glass or leaden cup. In one of the arms, *i*, is fixed a little glass tube through which the acid escapes. On the top of the framework of the tower a contrivance is fixed for maintaining the "wheel" in an upright position. As soon as the glass tube is filled with acid the "wheel" revolves regularly on its axis by the force of the acid escaping in a constant stream from the open tube. The "wheel" should stand exactly in the middle of the top of the tower. Each tubelet must thus receive precisely the same quantity of acid, whatever the number chosen.

Denitrating Tower.—The "denitrator" of Gay-Lussac's arrangement may be constructed in all respects similar to the absorber, but of much smaller dimensions, as 10 ft. high, and 2 ft. 6 in. diameter, for the same amount of sulphur converted daily. In this also any increase in size is recommended to be effected in height rather than in width.

The nitro-sulphuric acid derived from the "absorber" is conveyed by suitable apparatus to a vessel situated above the denitrator, whence it is admitted in a constant flow to the interior of the denitrator. In its descent through the tower it meets with the newly generated kiln gases which, accompanied by a certain amount of steam, are introduced at the bottom of the tower. The sulphurous gases appropriate the nitrogen compounds contained in the descending acid and carry them into the chambers, while the denitrated acid flows from the bottom of the tower into a suitable receptacle.

Conduct of the Process.—In order to ensure the conversion of all the sulphurous gas admitted to a chamber into sulphuric acid, it is necessary to have an excess of nitrous gas present. When an apparatus is employed which will recover all unproductive excess of nitrous gas, it may be supplied in such proportional superabundance as

may be considered beneficial ; but where no such apparatus is used the manufacturer must be content with losing a portion of the cheaper sulphurous gas.

We have already given some rough tests for the character of gases, &c., in this manufacture. In the pipes connecting the "absorber" with the chimney, and with the chambers, glass cases are sometimes inserted, as it is important to ascertain the nature of the gases before and after their passage through the tower. Their colour before should be yellow and afterwards white.

Sufficient nitre must therefore be used to maintain the escaping chamber gases at a yellow colour.

Smith's Remarks.—Referring especially to the absorber, Mr. H. A. Smith remarks that although the principle involved in the Gay-Lussac tower is very perfect, there are many practical difficulties in the way. In the first place it is very difficult to ensure the proper degree of strength being maintained in the acid supplied to the top of the absorber, as the workmen are very apt to neglect this precaution, and to use the first acid that comes to hand. Also the chamber draught requires very careful regulation, otherwise a large amount of the nitrous gases will escape the sulphuric acid in the tower, and make its way into the atmosphere as a loss.

Then again arises a difficulty in finding the requisite supply of nitric acid. It is, he thinks, impossible to have the theoretical amount of nitric acid present which is required to convert the total amount of sulphurous acid produced, so that manufacturers have to choose between two evils—allowing either sulphurous or nitric acid to escape.

McCulloch's Remarks.—McCulloch says that the acid used in the absorber should never be below 148° Tw. and as cool as possible, although were it even to be as high as 110° (? Fahr.) at the top of the tower, its absorbing power would not be very much affected, as the column is kept very cool

by the cold gases from the chambers. Acid admitted at 120° Fahr. at the top only showed 60° Fahr. when drawn off at the bottom.

The strength of the acid also is reduced in the passage from about 150° Tw. to 120° Tw.

Another authority estimates that 1200 pints of acid at 152° Tw. should be supplied to the absorber every 24 hours for every ton of sulphur converted in the same time.

Dr. Affleck's Remarks.—In the opinion of Dr. Affleck much nitre is lost because the Gay-Lussac absorber is not made with sufficient capacity for its purpose, when the dilute character of the gases is taken into consideration. He thinks that most of them should be doubled in *horizontal area*, and that they should not have a less capacity than 80—100 cubic feet for every ton of pyrites burnt weekly, and then with a chamber capacity of 3000—4000 cubic feet per weekly ton of pyrites, he would expect to work with $\frac{3}{4}$ —1 per cent. of nitre on the pyrites. While maintaining the height of the absorbing tower at its present standard of 40—50 ft., he would create additional capacity by an augmentation of the horizontal area, and with this advantage that the coke packing might thus be made closer without risk of choking the draught.

Since the absorption of the nitrous gas is more complete in proportion to the dryness of the gas, it is sometimes passed through or over a layer of concentrated sulphuric acid before entering the absorbing tower.

According to one estimate, 10 pints of thoroughly nitrated sulphuric acid will yield sufficient nitrous gas to be equivalent in value to 1 lb. of nitrate of soda.

Modifications.—The Gay-Lussac process has been very generally introduced, but many are the modifications to which it has been subjected at different times. The absorber has been the least modified, and principally in the following manner. In some manufactories, instead of having several small absorbers, one large tower of 14 lbs. (or

heavier) lead is lined with half-thick fire-bricks made to suit the curve, and its dimensions increased to 60 ft. in height, and 7—8 ft. in width.

Finkenhöher replaces the leaden tower by a wide stoneware cylinder made in several sections and fitted internally with alternating and inclined porous clay slabs, over which the concentrated acid falls, encountering in its splashes the ascending chamber gases. The porosity of the slabs is intended to have the same effect as coke.

But especially the denitrator has undergone many changes, and it is doubtful whether it would be possible to find an example of that tower as originally designed. Very great difficulty was experienced in finding any material or combination of materials to be used in its construction which were capable of resisting the great heat and intense chemical action which ensued when sulphurous acid was passed through nitro-sulphuric acid.

One suggested stoneware shelves as in the absorber. Another arrangement proposed consisted in placing a small stoneware vessel so as to form part of one of the sides of the chamber. The nitrous sulphuric acid is made to flow from a cistern into the vessel, its supply being regulated by a tap. A jet of steam is admitted from a steam-pipe through a rose at the end of the pipe inside the vessel, which decomposes the nitro-sulphuric acid. The gaseous nitrous products escape into the chamber through one pipe, while the sulphuric acid flows on to the floor by another.

Indeed, as Mr. Smith goes on to say, there have been some plans proposed which would in reality do away with the Gay-Lussac tower altogether, but these are, when inquired into, seen to be merely modifications of that plan. Of these, the most important, perhaps, was one brought forward some years ago by Mr. Hofmann, and which was tried in this country with very indifferent success, by Mr. Peter Spence, the great alum manufacturer.

Hofmann's Plan.—Mr. Hofmann's plan consisted of so

regulating the steam admitted to the first chamber as to produce acid at a specific gravity of 1.714 or 143° Twaddell, and he claimed to thereby render it "possible to manufacture sulphuric acid with a consumption of only 1 lb. of nitric acid per 100 lbs. of sulphur burnt."

Spence's Experiment.—Mr. Spence tried a modification of this plan by connecting three chambers, into the first of which he passed the sulphurous acid from the kilns, and working in the ordinary manner, obtained acid of 1.5 specific gravity = 100° Twaddell. He worked chambers 2 and 3, however, without steam, thus producing acid of 1.715 specific gravity = 143° Twaddell, which he ran back into No. 1, where, meeting with the weak acid, it parted with a considerable quantity of its nitric acid, thus reducing the amount of nitre required to be used.

Mr. Spence, writing in the *Chemical News*, vol. xxi. p. 189, thus describes his progress:—"When I commenced operations my consumption of nitrate of soda was exactly 4 tons 2 cwt. weekly. I have reduced it down to 3 tons, and my acid is still of good colour, showing sufficient nitric; and last week my production was rather over my average. I think I see my way to working with 2 tons nitrate instead of 4 tons 2 cwt."

We will quote Mr. Smith as to the final result of this experiment. The principal objection raised to this was the wear and tear the chambers were subjected to by the action of the nitric acid. In the two last chambers the action upon the lead must have been very great, as both the sulphuric and nitric acids must have been acting upon it. I must certainly also agree with Mr. Spence when he says " (in the same volume of the *Chemical News*) "that not only does he not believe in Mr. Hofmann's theory, but that 'the theory of sulphuric acid production, as well as the facts of the Gay-Lussac process, in my opinion, disprove Mr. Hofmann's hypothesis.'"

This is simply a modern confirmation of the old estab-

lished opinion that it is not economical to produce acid in leaden chambers at a high degree of strength, on account of the disproportionate wear and tear occasioned in them. The saving thus effected in nitre is counterbalanced by increased expenditure in another form.

The strength at which the chamber acid is maintained has much to do with the proportion of nitrate soda consumed, and therefore in comparing the relative advantages of nitrate soda in pots, nitrate of soda solution, and nitric acid, we must know not only the chamber capacity and monohydrated acid produce per lb. of sulphur converted, but also the strength at which the chamber acid is made, and which results from it, how long the chambers last. Without all these data no correct conclusion can be arrived at.

Steam Tower.—But to revert to the subject of denitration. A very important modification was the substitution of a steam tower, which has been worked by Mr. McCulloch with great success. It consists of an iron cylinder, which may be cast in segments and bolted together, 9 ft. high, and 3 ft. in diameter, varying in size according to the amount of work apportioned to it. This cylinder has an inner casing of stout lead to protect the iron from the acid and gases, and within this again is a lining of half-thick fire-bricks set in pipe-clay to safeguard the lead from the ill effects of the great heat and the sharp edges of the material forming the packing. The cylinder is set on end and packed with flints. A fire-clay pipe connects the top of the column with the chamber, and a steam-pipe is introduced at the bottom. The nitro-sulphuric acid from the absorber entering at the top trickles thence down through the interstices between the flints, where it is met by the ascending steam and reduced in strength from about 148° Tw. to 104° Tw., and raised in temperature from 60° to 300° Fahr., which on cooling down gives acid of about 128° Tw. almost perfectly free from nitrous compounds.

With this arrangement only $3\frac{1}{2}$ per cent. of nitrate soda was consumed on an average, using 25,000 tons of pyrites annually. It possesses the disadvantage, however, of requiring 3—4 tons of coal daily to boil up as much weak acid to 150° Tw., as is required in the absorber with the conversion of 12 tons of sulphur per 24 hours. Still Mr. McCulloch thinks very highly of the apparatus as having no liability to break down, and, in his opinion, admitting of a lower consumption of nitre than any other process.

In this last respect he found it even superior to the Glover's tower, after making many experiments with both. On starting a set of chambers with steam columns, the steam and acid were turned on 5 or 6 hours before charging the kilns, thus the chambers became filled with nitrous gases and steam ready to work when the sulphurous acid entered. Supposing the kilns to be charged at 6 a.m., it was found at 6 p.m., or 12 hours later only, that the drips were at 130° Tw., and the chambers quite ruddy, while on the following day the extra amount of nitre put in at starting could be dispensed with. With Glover's towers, on the other hand, it was found that at least 3 or 4 days elapsed before the tower became sufficiently hot to denitrate the acid, and that as no nitrous fumes were in the chamber to start with, there was a loss of sulphurous acid, and an extra consumption of nitre necessitated for a week at least.

Dr. Lunge says that Mr. McCulloch is the only man who has used steam towers, and that those which he worked at, Messrs. Allhusen's, have since been done away with and replaced by Glover's towers; that starting chambers with the latter is quite as easy as with the former, and that repairs are equally easy in both cases. As for the retention of nitrogen compounds in the Glover tower, which Mr. McCulloch fears, Dr. Lunge thinks that in this respect there is nothing to choose between the two plans, but that the Glover tower is perhaps a little the better in some

hands, even though it may not be quite so easy to denitrate the acid without diluting it. He does not hesitate in pronouncing the Glover tower as superior on the whole.

On the other hand, Mr. McCulloch rejoins that steam towers are in use elsewhere. He agrees that chambers can be started equally well *with extra consumption of nitre* in the case of the Glover towers, but that 10—12 per cent. instead of 3—4 per cent. will be necessary for days.

Glover Tower.—Through the great kindness of Wm. Glover, Esq., we are enabled to give the following details and figures concerning the Glover tower (fig. 48), a denitrating column which may be said to mark a new era in the manufacture of sulphuric acid.

The object of the tower is to denitrate and at the same moment concentrate both the chamber acid and the nitro-sulphuric acid from the Gay-Lussac absorber, thus effecting a triple economy, viz. in the consumption of nitre, in the consumption of fuel, for raising steam for the chambers and in plant, labour, and fuel necessary for concentrating chamber acid in leaden pans.

It should be placed between the kilns and the chambers' entrance, its size depending of course upon the quantity of acid being made.

The figure shows an oblong tower (this shape is preferred for convenience in construction) of sheet lead lined with hard siliceous fire-brick, and packed with flints, or flints and coke. The stream of sulphurous acid gas coming from the kilns enters at *A* below a perforated brick arch which carries the packing. In its ascent it meets constantly descending currents of mixed nitro-sulphuric acid from the Gay-Lussac or other "absorber," and ordinary chamber acid which have been previously admitted to the cisterns at the top of the Glover tower.

The sheet lead is generally about 14 lbs. per foot, but Dr. Affleck recommends that the side sheets up to the top of the kiln-pipe inlet should be of 30 lb. lead, and the bottom and turn

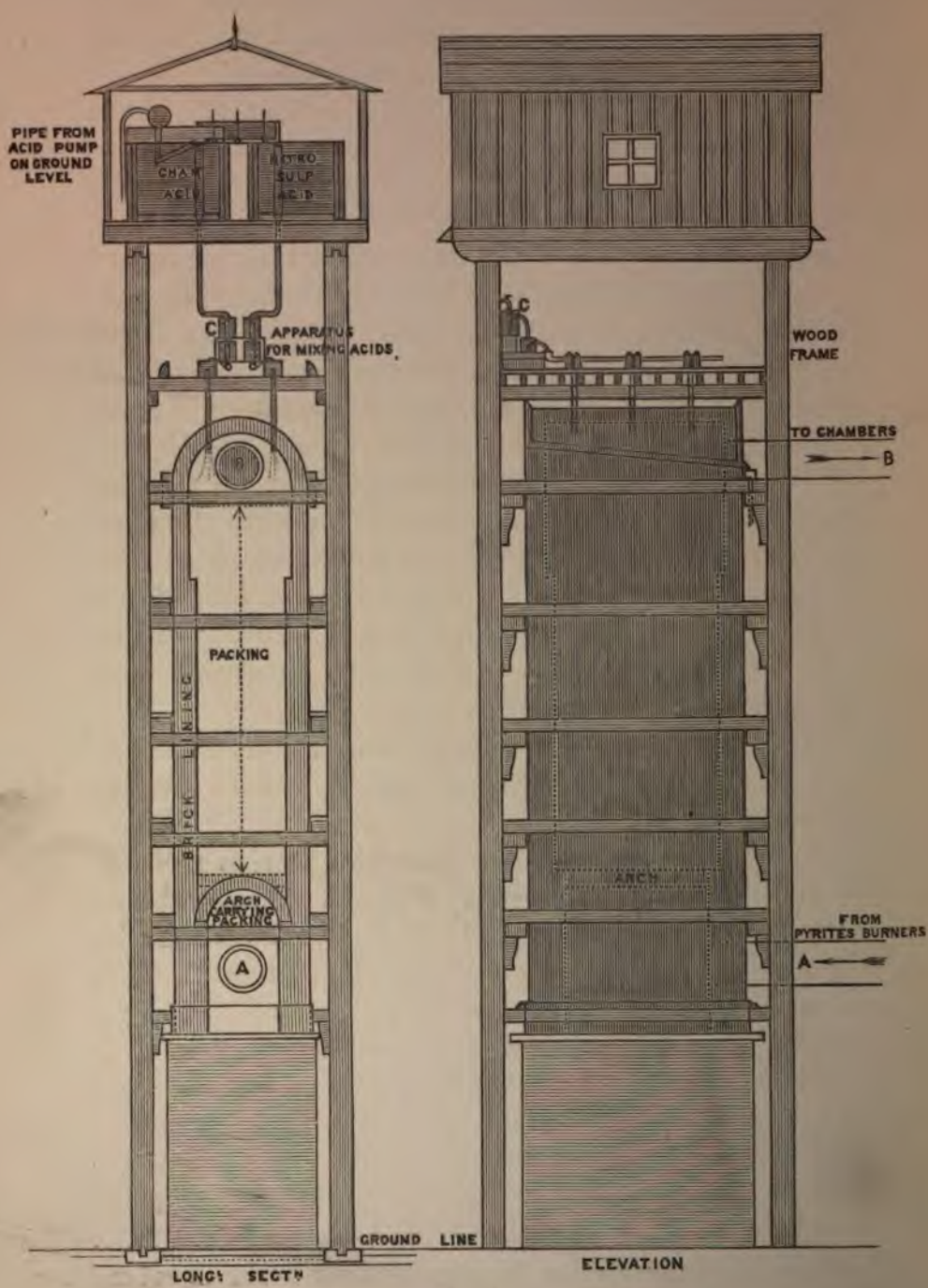


FIG. 48.

up of 50—60 lb. lead. He approves also of the height being limited to about 18 ft., instead of 25—30 ft., as in some instances, for in his experience the temperature of the gases entering the chambers from the Glover tower varies between 140° and 170° F., and he believes that a great height undoes to a certain extent what the lower part of the column has done. At least with the lesser height, better results are said to be obtained, especially in regard to the degree of concentration.

Replying to a reporter in the *Moniteur Scientifique* on the Vienna Exhibition, who said that the cost of erection and repair of the Glover tower was excessive, Mr. Glover states that he has a tower erected in January, 1863, at a cost of about 450*l.*, which has been in constant use till August, 1874, entailing about 70*l.* for repairs, and that during this time it has denitrated and concentrated more than 73,000 tons of acid up to 1.750 sp. gr. entirely by the combustion of 15,400 tons of pyrites. The tower was then still at work, and looked likely to last many years.

Fig. 49 shows the means adopted for conveying the requisite acid to and from the towers.

A is the Glover tower, *B* is the Gay-Lussac absorber. On the level of the ground are three tanks, *W* contains chamber acid, *S* concentrated acid from the outlet of the Glover tower, and *N* is supplied with nitro-sulphuric acid from the Gay-Lussac absorber. The outlets from each of these tanks, *W*, *S*, and *N*, communicate with the vessel *C* through a smaller vessel, *a*, which is fitted with three outlets, each furnished with a regulus plug, so that the vessel *C* may be supplied with acid from either one of the three tanks without interfering with the others. The vessel *C* is simply a boiler of 2 in. iron. When it is desired to fill the egg-shaped vessel *C*, one of the plugs is raised by the screw attached to it, and the acid flows in. As soon as *C* is full of acid the plug is screwed down and the engine *D* is set in motion. The steam cylinder is at *c*, and the air cylinder at *b*. Air is thus forced on to the surface of the acid in *C* until it

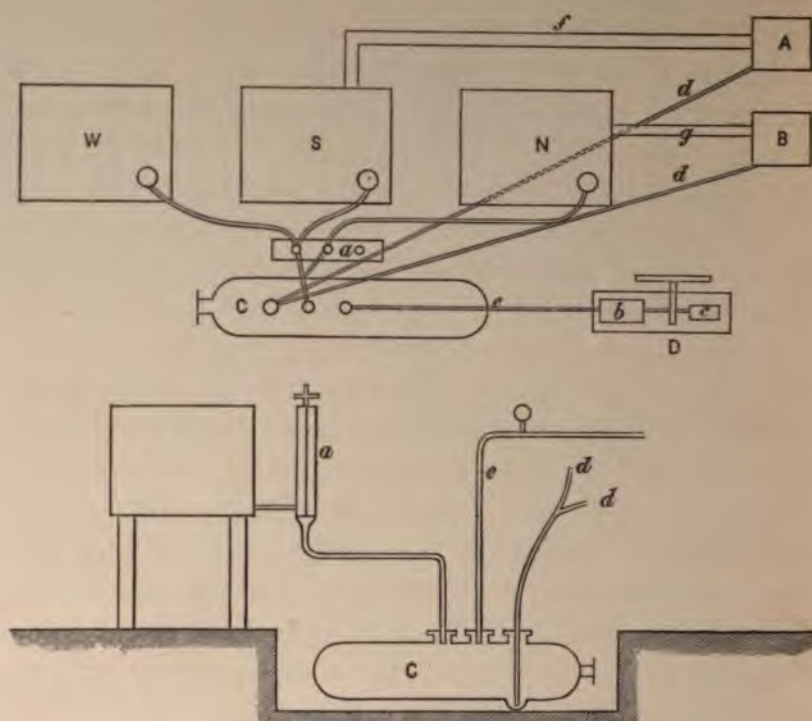


FIG. 49.

rises by means of one of the pipes, *d*, into the tanks prepared for its reception on the top of the towers. The air-pipe between the engine and *C* is provided with a gauge, and is marked *e*. The trough *f* conveys strong acid from the Glover tower to the tank *S*, while the trough *g* performs a similar service between *B* and *N*.

On the top of the Glover tower are two tanks for containing the chamber acid and the nitro-sulphuric acid which are admitted to the tower. The two acids are introduced by separate pipes into a distributing apparatus. For this purpose a "Barker's mill" is very commonly used. This apparatus is identical in principle with the Segner wheel already described, but, being entirely of lead, it is more easily and cheaply constructed, and answers the purpose equally well. Two of these "mills" are necessary on the Glover tower, one for each acid, as it is essential that the

acids should not mix until they have reached the interior of the tower. The tubelets admitting the acids to the towers are placed side by side in each instance, and fire-clay slabs are provided for the acid to fall upon, so that it may splash and spread as much as possible.

The reaction produced in the tower denitrates and at the same moment concentrates the descending acid, which is drawn off at the bottom of the tower, at a strength varying from 145° to 155° Tw., while the sulphurous acid gas, the disengaged nitrous gases, and the steam or vapour of weak sulphuric acid eliminated from the concentrated acid, pass together by the pipe B (fig. 48) into the chambers at a temperature of about 160° — 180° Fahr.

There can be no doubt that this tower will ultimately become as common an adjunct to a sulphuric works as the Gay-Lussac column. Certainly it is generally only used with pyrites kilns, because it is said that the combustion of brimstone does not afford sufficient heat to concentrate the chamber acid, though the denitration is performed with as great efficiency as in the case of pyrites. By using a brimstone kiln, such as Blair's, however, concentration can also be very considerably obtained. This has been practically proved by Messrs. Henry Glover and Co., and to the courtesy of these gentlemen are due some of the details concerning the conduct of the Glover tower.

Several authorities have found great fault with the Glover tower on account of the liability of the nitrogen compounds forming part of the kiln gases to become reduced to the lower oxides of nitrogen, and even to the element itself, by the great heat generated in the tower, and there is no doubt that with pyrites kilns this accident has sometimes occurred, but it may be remedied to a very great extent, if not entirely, by having the Glover tower of such abundant capacity that the sulphurous gas is well distributed.

Mr. McCulloch tried two experiments which proved, he

thinks, that some of the hyponitric acid is re-absorbed in the Glover tower, and that something more than heat is required to denitrate the acid.

Vorster thinks it is better to admit the nitro-sulphuric acid to the chambers after dilution with hot water, and only to use the Glover tower for concentration. His voluminous calculations in support of his project are proved, however, in the hands of Dr. G. Lunge, to be erroneous throughout, and there are very many well-regulated works in which the nitrous gases are admitted along with the sulphurous gases from the kilns into the Glover tower without fear of any important loss taking place.

F. Bode disapproves of Glover towers, so long as acid can be concentrated by the waste heat of the pyrites kilns, and Hasenclever says that with the tower it is impossible to prevent the solid pyrites dust from gaining admission to the chambers, but such trifling criticisms merit no refutation.

In reply to various objections, Mr. Glover writes, that though heat alone may not be sufficient to thoroughly denitrate the acid, still concentration by heat in an atmosphere of SO_2 , does it most effectually, and that so far from the acid not being denitrated, it often smells of sulphurous oxide. He has a tower which he used for 6 years in a chamber system converting 1651 tons of sulphur (from Norwegian pyrites) into acid, with chamber space of 20 cubic ft. per lb. of sulphur, and the nitre used was 63 tons 13 cwts. or 3.8 per cent. Further all the acid was concentrated to 140° — 150° Tw.

When the denitrated chamber acid is not required for use as fast as it is denitrated and concentrated in the Glover tower, it may be conveniently run back into the chambers, and afterwards used from them direct. Only a trifling loss of nitrogen oxides is thus incurred, as the absorbing powers of this low strength acid are not at all great.

Sources of loss of Nitrogen.—Mr. McCulloch says that the losses of nitrogen compounds, principally in (*a*) absorbed in the acid on the floors of the chambers, (*b*) escaping from the absorbing tower, and (*c*) remaining in the acid flowing from the denitrating column, will probably amount in the best-planned works to about 3—6 per cent. Hasenclever thinks that only one of the above sources of loss is worthy of consideration, viz. the reduction of the higher nitrous oxides in the Glover tower. He adds that when the proportion of free oxygen in the gases entering the chamber is too low, this reduction may take place, even without a Glover tower.

Davis's Remarks on Arsenic.—The following table and observations concerning the part played by arsenic in the loss of nitrous compounds, and gathered from a paper by Mr. G. E. Davis, cannot fail to be found extremely interesting.

In the following table *A* is a works having 6 chambers all in one series, and the exit from the last going direct to the chimney. The escape tests were taken during one week when the exit was sulphurous acid and the chambers working badly.

B refers to the same works when the chambers were going well and the exit was straw-colour.

In *C* there were 9 chambers worked in three sets of three each producing well. The exit gases were collected and passed up a tower 5 feet square and 30 feet high, filled with coke and furnished with a steam jet. The acid ran from it at about 70° Tw.

The gases were straw-coloured on entering, and colourless on leaving the tower.

There were four chambers in *D*, supplied from brimstone kilns. They were worked in a set, the gases entering No. 1, and passing from No. 4 direct to the chimney.

E possessed 6 chambers worked in one series, and in conjunction with two Glover towers and one Gay-Lussac

Exit gases tests.	Area exit pipe. sq. ft.	Speed, draught per second. cub. ft.	Nitrate soda used per week. cwts.	N ₂ O ₂ con- tained in nitrate soda. cwts.	N ₂ O ₂ escape as gas per week. cwts.	Acid used per week. ³ tons.	Con- taining N ₂ O ₂ per cent.	Loss of N ₂ O ₂ in acid per week. cwts.	Acid used in towers per week. ² tons.	Con- taining As ₂ O ₃ per cent.	Weekly As ₂ O ₃ cwts.	N ₂ O ₂ reduced to N ₂ O ₂ per week. ³ cwts.	N ₂ O ₂ imper- fectly absorbed cwts.	N ₂ O ₂ unac- counted for. cwts.
A. Average. ¹ $\left\{ \begin{array}{l} a. 22.0 \\ b. 2.5 \\ c. 24.5 \end{array} \right\} = 1.8 \text{ N}_2 \text{O}_3$	1.227	7.4	34.5	14.6	12.6	50	.126	1.26						.74
B. $\left\{ \begin{array}{l} a. 1.16 \\ b. 2.52 \\ c. 3.68 \end{array} \right\} = 1.8 \text{ N}_2 \text{O}_3$	2.6		50.0	21.2	19.4	90	.042	.756						1.044
C. $\left\{ \begin{array}{l} a. 0.22 \\ b. 1.50 \\ c. 1.72 \end{array} \right\} = 1.07 \text{ N}_2 \text{O}_3$	.884	12.0	28.0	11.87	8.24	45	.3	2.70						.93
D. $b = 1.5 \text{ N}_2 \text{O}_3$	1.00	6.4	20.0	8.50	7.40	40	.04	82	140	.35	9.8	7.52	1.66	.78
E. $b = .35 \text{ N}_2 \text{O}_3$	4.00	8.5	21.0	8.925										
F. Wanting	4.17	12.0	32.0	13.6		100	.05	1.00	110	.7	15.4	11.82		.78
G. $b = .28 \text{ N}_2 \text{O}_3$	5.00	16.0	42.0	17.8					120	.4	9.6	7.3	6.88	3.62

Remarks.—¹ a. = Grains Na₂ CO₃ neutralized by SO₂ from 1 cub. ft.
 b. = " " "
 c. = " " "
² Acid calculated at 123° Tw.
³ According to the Equation As₂ O₃ + 2 N₂ O₂ = As₂ O₅ + 2 N₂ O₂.

absorber. They used Tharsis copper pyrites and were doing well.

The four chambers composing *F* were very badly arranged. They were worked with one Glover tower and one absorber. No. 1 chamber was placed at a higher level than the others, and the acid formed in it was run into No. 2, which was often highly charged with nitre. All the acid was not run down the Glover concentrating tower, but much of it was drawn off directly into the pans. Also an undue proportion of concentrated acid was used in the absorbing tower.

Finally, the chambers of *G* were old, their production low, and their working very variable. All the acid was passed through the concentrating (denitrating) tower, and contained only a trace of N_2O , when used.

In the case of the four first systems, *A*, *B*, *C*, and *D*, which were worked without towers of any kind, it will be seen that the losses of N_2O , taking place in the escaping chamber gases, amount respectively to the proportions of 86.3, 91.5, 69.4, and 87.0 per cent. of the total.

Attributing the loss of N_2O in the systems *E*, *F*, and *G* to arsenic, the following table has been drawn up from the data above given, which shows in column—

1. The number of cubic feet of air passing the exit per ton of acid at 123° Tw. per week.
2. The percentage of nitrate of soda rendered useless by arsenic.
3. The percentage run away in the chamber acid, and in
4. The lowest percentage of nitrate of soda on pyrites burnt, disregarding all loss but No. 2.

	1.	2.	3.	4.
<i>E.</i>	126,933	81.9	traces	1.03
<i>F.</i>	302,400	87.6	7.3	2.80
<i>G.</i>	336,000	41.0	traces	1.13

Thus, it is argued that if it be desired to reduce the consumption of nitrate of soda, the acid used in the absorbing tower must be free from arsenic. Judging from the figures shown by *E*, Mr. Davis thinks that the nitrate of soda proportion might be reduced to .26 per cent. on the pyrites burnt, or to 2.91 lbs. nitre per ton of acid at 123° Tw.¹

These observations have, however, been severely criticized by Mr. McTear, who considers, among other things, that it is difficult to prove that nitrous compounds enter the chambers from the Glover tower as N_2O , and thinks that it is certainly only partially true that the arsenic which is present in the nitro-sulphuric acid is at the same time reduced to arsenious acid, and as such leaves the concentrating or denitrating tower, for he has found arsenic as well as arsenious acids in all the Glover tower acids which he has examined. Again, he believes it not proven that the reaction in the Gay-Lussac absorber is an oxidation of As_2O_3 by the N_2O present, N_2O escaping in proportion, and that the amount of As_2O_3 is the measure of the limit of nitre that can be used. In the first place he denies that all the arsenic present in the nitrous sulphuric acid is as arsenic acid, and in the second place he is not certain that it is N_2O which oxidises the As_2O_3 , especially in the presence of higher oxides of nitrogen, which are always to be met with in Gay-Lussac acid. Finally he condemns all the calculations as offering no guide because they are based on the examination of 1 foot of gas three or four times in a week, whereas from 20,000,000 to 48,000,000 of feet of gas are escaping weekly, and under very various conditions of draught, &c.

¹ We have just met with some observations by Mons. M. E. Hjelt on the subject of arsenic in sulphuric acid, which are perhaps best inserted here. He found in chamber acid .202, in Glover tower acid .331, and in Gay-Lussac acid .334 per cent. of arsenic, the bulk of which was present as arsenious acid. In the last chamber the acid contained merely .019 per cent. of arsenic. The mud deposited in the Glover tower he finds to consist principally of arsenious acid.

Concerning these criticisms, Mr. Davis has replied, that in the case of the four chambers without towers (*A, B, C, D*) he has accounted for nearly all the nitrate of soda used. That with the chambers having towers he does not claim to have proved where the loss of nitrate soda takes place, but he argues that a loss does occur by the reaction of arsenious acid on nitrous anhydride from the observation that the arsenic acid is produced in the tower from the arsenious acid which the acid contained and from finding nitric oxide in the escaping gases. It has been pointed out to him that the arsenious acid was most probably oxidised by N_2O_4 ; if so, then another source of loss must be sought, but he has pointed out that the loss of N_2O_3 is nearly equal to the amount which would be decomposed by the arsenic run down the absorbing tower—a strange coincidence—and he has also observed in one works, that when the amount of arsenic in the acid was large, it was impossible to work with the minimum quantity of nitre used by other manufacturers.

Once more, Mr. McTear reiterates that Mr. Davis has not proved anything, as his observations were confined to such a small proportion of the escaping gases whose variation is very great. Also he has not taken into account the stock of acid in the process and its variation in percentage of nitrous compounds.

Further, Dr. G. Lunge intimates that without correction for temperature and barometer an error of 10 per cent. would frequently occur (at Zürich for instance) and complains that Mr. Davis has not made any test with pure substances nor with those containing known quantities of impurities.

Sulphuric acid manufacturers would, we venture to suggest, have better cause to be thankful to these critics, had they adduced some *proof* of Mr. Davis's error instead of heaping wholesale doubt and condemnation upon his researches. At present neither side has *proved* anything,

and we must await further evidence before this important question can be considered as decided. We hope Mr. Davis will not rest content with his past labours.

Methods of estimating Nitrogen in Nitro-sulphuric Acid.

—Various are the plans and means proposed for estimating the amount of nitrous compounds contained in a sample of sulphuric acid, the principal of which we shall give. As to their accuracy, there is great diversity of opinion, one chemist pronouncing in favour of one plan, another advocating another plan. One high authority goes so far as to say he places no dependence whatever upon analyses as a guide on this point. He has repeatedly tested nitro-sulphuric acid, and always found the test to contain over 6 per cent. of NaONO_2 , i. e. 31.49 lbs. of NaONO_2 , which, added to 3.5 per cent. of nitrate of soda used from the store, is equivalent to 23.5 per cent. of nitre on the sulphur charged. As he proved it to be impossible to reduce the amount taken from the store, he can only conclude that the analyses are futile, and he prefers to judge of the character of the acid by such simple tests as those already indicated, viz. colour, smell, frothing, &c.

One plan of estimating the degree of absorption of nitrous compounds in the acid is as follows. A tube of 20 cubic centimetres is filled with a salt solution of 100° Gay-Lussac, and its contents poured into a vessel containing about $\frac{3}{4}$ pint of distilled water. Another instrument is supplied with 20 cubic centimetres of the nitro-sulphuric acid to be tested. This should be graduated in 200 divisions, and the acid poured from it into the salt solution in the most gradual manner; until by the addition of a few drops of sulphuric acid solution of indigo the blue colour is no longer destroyed. The more acid required to destroy the colour of the indigo, the less nitrous compounds it contains. If 17—20 parts only have been used, it is roughly estimated that 10 pints of such acid returned to the chambers and properly denitrated will do the work of 1 pound of nitrate of soda.

As a check upon the action of the denitrator, a drop of indigo solution may be put into a sample of the denitrated acid, when, if the colour be not destroyed, the nitrous compounds have been completely eliminated from the acid.

Hart's Method.—Hart's method of estimating the nitrous sulphuric acid is well known. He avails himself of nitrate of urea, as it is easily procurable in a pure condition. Placing 20 grains of this in $2\frac{1}{2}$ oz. of water, he raises the solution to a boiling temperature. A white plate or other convenient vessel is covered with some drops of a solution of potassic oxide in thin starch water. The nitro-sulphuric acid is supplied from an alkalimeter to the urea nitrate solution, and kept constantly stirred, continuing the supply until a drop mixed with the starch solution creates a blue colour by liberating the iodine, thus indicating the presence of free nitrous acid. The calculation may then be made upon the basis that 12.35 grains of nitrous acid will decompose 20 grains of nitrate of urea. This amount of nitrous acid is reckoned as equal to 27.64 grains of nitrate of soda.

Crowder's Methods.—Crowder proposes the subjoined plan. The bottle of the instrument, which is much like Geissler's carbonic acid apparatus as described by Fresenius, is filled to the extent of one half with water containing about 20 grains of urea or 30 grains of nitrate of urea in solution. The instrument is weighed and the acid tube filled with the nitrous sulphuric acid to be tested, and weighed again. The difference is the weight of the substance taken. The tap allowing the exit of the nitrous sulphuric acid is opened, and a slow stream of acid falls down through the solution of nitrate of urea to the bottom of the vessel. In a few minutes the acid tube becomes empty, the apparatus shaken to cause complete admixture, and, after the violent effervescence that at first takes place has subsided, the instrument is heated to about 200° to separate the dissolved gas. When cold it is sucked through to remove the gas remain-

ing in the apparatus, and weighed. The difference in weight before and after the operation indicates the quantity of carbonic acid and nitrogen liberated. Urea decomposed by nitrous acid gives 2 equiv. Carbonic acid, 4 equiv. Nitrogen, and 4 equiv. water. He takes about 300 grains of the nitrous sulphuric acid.

$C_2N_2, H_2O_2 + 2 NO_2 = 2 CO_2 + 4 N + 4 HO,$
therefore

$$2 CO_2 + 4 N : 2 NO_2 \text{ quantity } CO_2 + N \text{ found,} \\ \text{Equiv.} = 100 : 76 : x :: \text{nitrous acid found.}$$

He thinks the effervescence in Hart's test may cause much error by loss, and finds that iodine does not manifest its reaction so soon at one time as at another; hence the superiority of gauging the amount of gas evolved.

For comparison another plan was tried with a solution of permanganate of potash in water of such a strength that a burette divided into 1000 grs. measure of water oxidised a solution containing about 5 grs. of iron to peroxide, as in Margueritte's method. To peroxidize protoxide of iron requires the addition of 1 equiv. of oxygen to every 2 equiv. of iron, therefore

$$\begin{array}{l} \text{If 2 Equiv. Fe : 1 Eq. O :: Fe : O} \\ 56 : 8 :: 5 : .714 \end{array}$$

In the reaction which takes place when permanganate of potash is added to nitrous sulphuric acid it is assumed that the whole of the nitrogen compounds present are in the form of NO_3 . This is in accordance with the experience of those who have paid any attention to the subject. It is further assumed that this NO_3 on the addition of permanganate of potash is converted into nitric and not hyponitric acid.

The above .714 of oxygen would therefore represent the following quantity of NO_3 :—

$$\begin{array}{l} 2 \text{ Equiv. O : } NO_3 :: O : NO_3 \\ 16 : 38 :: .714 : 1.7 \end{array}$$

The process is conducted thus. About 100—150 grs.

of nitrous sulphuric acid to be tested is weighed out in a beaker of some 6 ounces capacity. The standard solution of permanganate is poured from the burette, and the number of graduations noted at the point at which the acid ceases to decolorize the permanganate. Care must be taken at the commencement of the operation not to shake the acid and solution, and to keep a little permanganate floating on the top of the acid until the latter is somewhat diluted, otherwise there is a fear of losing a small quantity of nitrous acid by effervescence before it becomes oxidized; this can, however, be very easily managed with ordinary care. The results are said to be perfectly constant, to illustrate which a number of tables are given (*Chemical News*, vol. xxiv. p. 249).

The operation may be varied with advantage for manufacturing purposes by taking a measured quantity of standard solution of permanganate, and dropping some of the nitrous sulphuric acid to be tested into the solution by means of a graduated tube with a pinch cock, the advantage being that much less time is required, and that being done by measure no trouble nor time is spent on weighing. This method has been for some time in very successful operation in some large works.

From some cause which, as yet, Mr. Crowder is quite unable to account for, the results obtained by this process are about one-third less than those obtained by the urea method. Whether there is any inaccuracy in the way of calculating and comparing the results he cannot yet discover, but by taking sulphuric acid containing a known quantity of nitrous acid as determined by the urea process, and standardizing the permanganate by that quantity, results were obtained agreeing perfectly with the quantity of NO_3 in other samples as determined by the urea method.

The tables given show :—

1. The constancy of results obtained by the processes adopted.

2. A series of analyses of samples of nitrous and denitrated acid from four different works, illustrating the three different methods of denitrating, viz.

- a. By steam alone.
- b. By a mixture of steam and sulphurous acid.
- c. By passing nitrous sulphuric acid down a column (presumably a Glover tower) and denitrating and concentrating it at the same moment by a current of hot sulphurous gas as it comes from the kilns.

Dr. Lunge's Methods.—Dr. G. Lunge believes that many discrepancies arise between the various results of analyses of nitrous sulphuric acid because they are not controlled in many cases by means of a substance of absolutely certain composition. As such he employed silver nitrite, obtained by mixing a hot solution of silver nitrate and sodium nitrite. It was recrystallized twice from boiling water, and dried *in vacuo* over sulphuric acid. Several test liquors were made of artificial nitrous sulphuric acid. Each time 5 grammes of pure Ag. NO_2 were dissolved in 500 Cc. pure concentrated sulphuric acid 1.842 sp. gr. at 15°C. , in such a way that the salt only came into contact with the acid at the bottom of the vessel, so that the N_2O_5 evolved was immediately dissolved by the acid, and only one or two bubbles of it escaped. The sulphuric acid employed was absolutely free from nitrogen compounds, as was proved by the most delicate of all re-agents, diphenylamine. The liquid obtained was perfectly clear, the silver sulphate dissolving in the strong acid.

This liquid was used for testing with permanganate of potash, and with bi-chromate, also with urea. Further, the silver nitrite was tried directly by reduction in an alkaline solution (Siewert's process). It should be stated from the outset that the only really accurate method of estimating N_2O_5 was proved to be that by means of potassium permanganate. But this plan also is only perfectly accurate when used in the less convenient form of running the nitrous

sulphuric acid from a burette into a measured quantity of permanganate solution, so as to oxidize the N_2O , momentarily, before it can split up into NO and NO_2H (with the water present) since a certain quantity of NO always escapes if the opposite plan be followed. Very many tests were made in all ways and the last named was the only one which gave really accurate and wholly constant results.

The permanganate solution was employed as a semi-normal one, that is to say giving up .004 oxygen for each Cc. or indicating .0095 N_2O_5 . It had to be diluted pretty strongly, say 1:20 water, to avoid too great a heating. When the temperature of the measure rises above $80^\circ C.$, the results are no more accurate, from 30° to $40^\circ C.$ is the most convenient temperature, as then there is no danger of overheating and at the same time the reaction takes place more rapidly than in the cold. When ordinary nitrous sulphuric acid 1.700 sp. gr. is used the rise of temperature caused by its mixing with the test solution is only slight, and it is advisable to dilute the permanganate with tepid instead of cold water in order to hasten the reaction.

Potassium bi-chromate and bleaching powder are alike used, but neither will bear comparison with the permanganate.

Siewert's method (reduction by zinc and iron in an alcoholic solution of potash) was also tried, but as no more accurate results were obtained with silver nitrite than with potassium nitrate, this tedious plan was at once abandoned.

Very careful attention was given to an examination of Hart's urea method, but the reaction was found to be by no means so simple as Hart supposed. Much ammonium salts are formed.

Crowder's urea test was found superior to Hart's, but it possesses an objectionable feature in the necessity of weighing a heavy apparatus three separate times in a delicate balance.

Altogether it seems to be established that the permanganate of potash test is undoubtedly the best, and that when conducted with the simple precautions already hinted at its results are perfectly reliable.

It is also confidently affirmed by Dr. Lunge that no nitric acid (or the very faintest traces only) is found in nitro-sulphuric acid, though it may sometimes be generated during the testing process, which, however, cannot happen with the permanganate method.

Escaping Gases Tests.—In the great majority of sulphuric acid works the product is calculated in a very rough-and-ready manner, and no attempt is made to keep an exact nor even an approximate record of the amount of gas which escapes from the chamber system into the atmosphere. The attention of manufacturers in this country has recently been more particularly attracted to this subject on account of the Noxious Vapours Commission, and probably within a few years the law will be so shaped that economy will be enforced, by the escape of the gases being restricted under penalty to a very low figure.

Mactear's Apparatus.—The transactions of the Newcastle-upon-Tyne Chemical Society (Vol. iii. part xiv.) contain a paper from the pen of Mr. James Mactear on this subject, which is accompanied by diagrams, &c., illustrating the very laborious and profound experiments which he conducted through a series of years upon the estimation of the amount of sulphuric acid constantly escaping to the atmosphere.

He gives a useful formula for calculating the available sulphur in a pyrites, thus:—

$$\left. \begin{array}{l} \text{Percentage of sulphur} \\ \text{in pyrites.} \end{array} \right\} - \frac{\left. \begin{array}{l} \text{Percentage S.} \\ \text{in residue.} \end{array} \right\}}{100} \times \left\{ \begin{array}{l} \text{Weight of residue} \\ \text{per cent.} \end{array} \right.$$

Taking, for example, a pyrites containing 47.5 per cent. of sulphur, and supposing that 100 lbs. of these pyrites yield 70 lbs. of waste ore or cinders containing 3.57 per cent. of unconsumed sulphur, we get—

$$47.5 - \frac{(3.57 \times 70)}{100} = 45 \text{ per cent. of sulphur extracted.}$$

This method of estimating is far more correct, of course, than when the percentage of sulphur contained in the cinders is simply subtracted from the original percentage before calcination, as the difference in weight has to be taken into consideration.

The gas-testing apparatus mainly consists of a set of absorbing tubes and an ordinary gas meter, both of which are kept in locked cupboards.

For gauging the escape of oxygen, two earthenware jars are used, one placed higher than the other, and the upper, filled with a solution of common salt or with water, empties into the lower, thus creating a vacuum. The tubes are so arranged that the inlet for the gases aspirated goes to the bottom of the jar, as also does the run-off pipe, so that the head of water in each case is equal, and the rate of flow nearly constant. It may be left for any length of time, from 1 to 24 hours, and a sample of the aspirated gases contained in the upper jar may then be tested for oxygen, for which purpose he prefers an ammonia solution of chloride of copper.

The measuring-tube consists of a bulb and stem, the latter holding 21 per cent. of the whole bulk, and so divided that 20 per cent. can be read off with ease.

The amount of oxygen escaping from chambers varies considerably. In France and Germany it is kept as low as possible. A fair allowance will perhaps be 5 per cent. by volume of the total escape when brimstone is used, and 6.4 per cent. when pyrites are employed as a source of sulphurous acid. It often amounts to 10 per cent. of the volume leaving the Gay-Lussac tower.

Mr. Mactear suggests that the loss of gas taking place should be stated as sulphuric acid, and not as sulphur. Thus, for example, when the oxygen is 10 per cent. and the

Ba SO₄ found in .12 grammes per cubic foot we have the calculation :—

$$\text{Ba SO}_4 = .12 \times 15.06 = 1.807 \text{ H}_2 \text{ SO}_4 \text{ on 100 sulphur burnt.}$$

If the oxygen be 8 per cent. we have $1.807 \times .8437 = 1.52$ per cent. of sulphuric acid on the sulphur burnt.

The returns can then be made up thus :—

H ₂ SO ₄ actually obtained	
„ lost as found by the above test	
„ „ in cinders as unburnt sulphur	
„ unaccounted for	
	<u>306.25</u>

In the discussion which followed the reading of Mr. Mac-tear's paper, the opinion seemed to be pretty general that his calculations were based upon a somewhat arbitrary ground, and the irregularities consequent upon charging of the kilns, alterations in the rate of the draught, &c., would altogether vitiate them.

Glover's Apparatus.—By the courteous kindness of W. Glover, Esq., of Wallsend, we are enabled to give the annexed sketch of the very simple apparatus designed by him for the estimation of the amount of acids escaping in every cubic foot of atmosphere passing from the chambers into the chimney, and which is found to give such perfect results as to leave nothing to be desired.

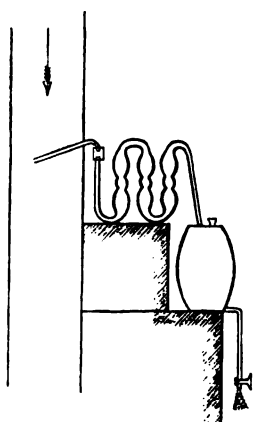


FIG. 50.

It consists of a barrel (fig. 50) made to contain an accurately measured quantity of water, which may vary from 1 to 12 cubic feet, according as it is desired to take occasional tests only or to make a constant estimation of the escaping gases over a period of twelve hours, as for instance during the night. This barrel is fixed in a suitable manner in close proximity to

the flue which carries the gases escaping from the Gay-Lussac absorber to the chimney.

Into this flue is fixed a glass tube, projecting sufficiently into the interior of the flue to meet the full volume of the gases, and inclined downwards a little to avoid the accidental entrance of liquids. To this glass tube are connected by means of removable India-rubber tubing a set of three glass bulb tubes, the first two of which are filled with a sodic solution of known strength, while the third contains pure water coloured with litmus. The last tube is in communication with the barrel. When the barrel has been accurately filled with water, and the connexions between the various parts of the already prepared apparatus have been properly made so as to produce a perfectly air-tight arrangement throughout, the cock terminating the pipe leading from the bottom of the barrel is opened. Thereupon the water will commence to escape from the barrel, producing a vacuum in the upper part of it, which exerts a suction upon the flue gases, and draws them into the barrel to supply the place occupied by the water. This will continue as long as the water runs from the barrel, and until the barrel lately filled with water has instead become full of air. The rate of the exit of the water from the barrel must therefore be regulated to suit the length of time during which it is intended that the test shall be carried on.

In the passage of the gases through the apparatus as indicated, the acid portions of them will be arrested by the soda solution, and if that solution be in sufficient strength and quantity, all the acid gases will be so arrested, and nothing but air will pass into the barrel. The third tube, containing the litmus-coloured water, is for the purpose of ascertaining whether the absorption of the acids be complete or not. In the latter case the liquid in the third tube will become changed in colour to a red tint, and it will then be known that the soda solution was not in sufficient force to fulfil the work required of it, and consequently the test

must be disregarded, and a fresh one made with a stronger solution.

All that remains to be done when a test sample has been procured, is to ascertain the amount of soda neutralized, and from that can be calculated in a few moments the exact proportion of acid contained in the volume of air which has been aspirated into the barrel.

The apparatus offers such a simple, cheap, and accurate method of ascertaining the amount of acid escaping into the atmosphere, that no manufactory should be without it. Of course it is not intended to abate or prevent the escape, nor to render unnecessary a careful watching of every chamber in the system, but it affords an unerring check upon the ordinary rough methods of judging of the degree of perfection of the process going on in the chambers.

Coquillon's Gas Tester.—At a recent meeting of the Tyne Chemical Society, Dr. Affleck referred in terms of high praise, to an instrument invented and patented by Mon. Coquillon, which will determine at the same moment the amounts of oxygen, carbonic oxide, carbonic acid, hydrogen, and nitrogen contained in a gaseous mixture. It is made by Abvergniat Frères, Paris.

Preventing Nuisance.—According to Dr. Letheby, it is the practice at some works to pass all the chamber gases that escape from the absorbing tower through a lime purifier, which thus prevents the exit of noxious vapours into the atmosphere. Of course it is not destined to effect an economy of the waste gases, but simply to avoid the nuisance which they create, and the mischief which they inflict upon neighbouring vegetation.

Regulation of Draught.—The regulation of the draught of the chamber system is best achieved by means of a sheet lead damper fitted into the flue which leads from the absorbing tower, or from the last chamber, if there be no absorber, to the chimney. The speed of the draught is a point which needs the most careful attention, in order to

keep the chambers in proper working condition, and instead of having anemometers and a variety of delicate apparatus for measuring the exact number of cubic feet of air which enter the kilns, it will be found far easier to check irregularities of draught, by adjusting the damper, for when once the chambers have become packed with gas, the opening of the kiln doors even will not admit a rush of air unless the damper be open wide enough to give it an exit at the other end. Our experience coincides with Mr. McCulloch's, that the best point at which to keep the draught is so that the gases, on opening the kiln doors for charging, show indecision whether to come out at the front or to remain in. The damper should be placed within a box under lock and key, so that it may not be trifled with nor regulated by the night kiln men to accommodate their drowsiness.

CHAPTER V.

CONCENTRATION.

WE have already seen that the strength at which acid is produced in the leaden chambers is generally about 113°—120° Tw. It is not economical to make acid in leaden chambers at a higher strength, for not only is the wear and tear of plant disproportionately increased, but also the greater the density of this acid the more sulphurous and especially nitrous gas is absorbed by it and thus wasted. This latter evil is abated, however, if all the chamber acid be put through a Glover tower.

Acid of this grade may be conveniently used, direct from the chambers, in the manufacture of artificial manures, nitric acid, &c. But there are other substances, such as alkali, whose production is often associated with sulphuric acid works, which require a stronger acid than that made in the chambers, and this must be procured by submitting the chamber acid to a process of concentration. Again, when the acid is intended for sale and transport outside the works, it is almost always concentrated, either partially or completely, in order to save the unnecessary carriage of useless water.

This concentration is performed in two stages. First the chamber acid is distilled (where the Glover tower is not in use) in leaden pans, to a degree of about 140°—150° Tw., at which point it is sometimes sold as “brown oil of vitriol,” and, if necessary, it is then transferred to other vessels either of glass or platinum, and the remaining superfluous water is driven off as far as it is possible

by the simple application of heat, that is to say until the acid has a density of 169° — 170° Tw., at which point it contains about $18\frac{1}{2}$ per cent. of water. For ordinary purposes this is the greatest strength required, but as a special demand sometimes exists for perfectly anhydrous acid, a description of the apparatus employed in its manufacture will be found at the end of the volume.

Leaden Pans. Surface-heated.—The form of pan commonly used by manufacturers who utilize their own concentrated acid is that called a "surface concentrator." The flame, &c., proceeding from the furnace is made to pass over the surface of the acid in the pan, and thence descends into the flue, carrying with it the water which it has evaporated. The lead being very liable to fuse at a comparatively low temperature, is enclosed in brickwork to protect it from the action of the flame. The side of the pan is brought up to the level of the top of the retaining wall, and within the pan is arranged a close row of fire-bricks on end, but not cemented in any way. Thus the sides and ends of the pan are enclosed between brick walls, and all that remains is to cover over the space between the walls, which is done by placing fire-bricks on the top, resting partly on the retaining wall, and partly on the row of bricks just mentioned; the flame is thus completely kept from the lead.

Some manufacturers employ a slightly different arrangement, although identical in principle. A covering brick is employed, made expressly for the purpose of the proper size, with an indentation or channel formed in it for the enclosure of the upper edge of the leaden pan.

Occasionally the extra precaution is taken of running a stream of cold water round the edge of the pan by means of a leaden channel which is burnt on to the side of the pan by the autogenous process.

Under-heated Pan.—Only acid intended for use by the manufacturer can be concentrated in this description of

pan, as the impurities carried into the acid by the passage of the flames over its surface render it so black and turbid as to make it practically unsaleable. In order to produce a clean marketable article (whether it be sold as brown oil of vitriol or refined to a higher degree), a pan heated from beneath must be used.

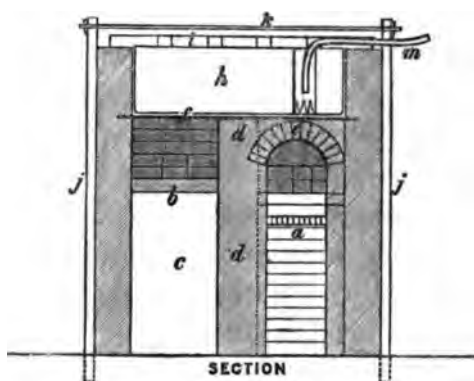


FIG. 51.

The figure 51 shows the arrangement and construction of such a pan.

a furnace bars.

b flue bed, formed of bricks.

c earth.

d side wall of flue, and central wall of support to the pan.

e furnace arch of fire-bricks: the sides of the fireplace are also of fire-brick.

f the cast-iron plates, about $\frac{3}{4}$ in. thick, covering the flue, and forming the bed on which the pan rests. These are rebated on both edges so as to make a close joint that will not admit the fire.

h leaden pan of 14 or 21 lbs. lead; the cover is of 4 lbs. lead, and is provided with a leaden pipe to carry off the vapours generated in the pan.

i pieces of wood to which the cover is attached by leaden straps.

j k cast-iron bars, and wrought-iron tie rods to strengthen brickwork.

m leaden pipe, $1\frac{1}{2}$ in. bore, through which the pan is fed direct from the chamber.

n (fig. 52) leaden syphon, $1\frac{1}{2}$ in. bore, through which acid is drawn from the pan.

o leaden cup, 18 in. deep, 4 in. diameter, attached to a weight by a chain passing over a pulley. This cup is filled

with acid, and the syphon is also filled with acid and set with one leg in the pan and the other in the cup. When the cup is lowered the acid flows through the syphon and overflows the cup, running into

p a leaden box, 3 ft. 3 in. deep, and 9 in. diameter, from whence it flows through

q a leaden pipe leading to cooler or retorts.



FIG. 52.

When the cup is raised so much that the top of it is above the level of the acid in the pan, the acid ceases to flow. In the drawing the cup is shown raised to its highest, the top being a little above the level of the top of the pan, so that were the pan quite full of acid, none would run out until the cup was lowered. The cup keeps the syphon constantly set, but if all the acid were drawn from the pan, air would enter the pan leg of the syphon, and it would become unset. Similar syphons are used for drawing acid out of the chamber.

The projecting corners of the leaden pan are not shown. The bottom and sides of the pan are formed of one piece of stout lead without cutting or joining, and the sides being bent up into place cause projecting corners.

A place where a leaden cup may be lowered to take a sample of acid to have its strength tried from time to time is also provided in the leaden cover and is closed by a luted cap when not in use.

This is a small pan capable only of concentrating about $1\frac{1}{2}$ tons per 24 hours, but much larger pans are in common use. Heat is frequently economized by placing two or even 3 pans in juxtaposition, arranging them one higher than another in such manner that the acid can be syphoned from one to the next, and only 1 fire is used, that being under the lowest of the series.

Purification of the Acid.—Should the acid in the pan be of a very dark colour (owing to the carbonization of foreign

organic matters which have found their way into it), its sale may be spoilt to some extent, and the colorization should be removed at this stage by introducing a little nitre or saltpetre into the boiling liquid in the pan. Sometimes also the acid has absorbed a quantity of nitrous gas, which though not detrimental to glass retorts, exercises a most injurious effect upon platinum, and must be removed before the acid is admitted to the still. It can be eliminated to the last trace by putting sulphate of ammonia to the extent of from 0.05 to 0.1 per cent. of the acid (according to circumstances) into the pan, but in this case, of course, the nitrous gases are lost. A far more economical plan consists in having a supplementary shallow pan where the chamber acid may first be heated by the waste heat from the concentrating pans and the retorts, and provided with a raised leaden cover burnt on all round, and having curtains depending from it. At one end a stream of hot sulphurous gas is introduced from the kilns and made to circulate over the surface of the heated acid by means of the curtains, taking its exit at the other end of the pan, which should have connexion with the "working" chamber. A great proportion of the oxides of nitrogen arising from the heated acid will be attacked by the sulphurous acid, and the sulphuric acid resulting from the reaction is collected in the pan, while that which remains of the gases passes into the chamber and is there utilized. Thus two economies are effected at the same moment.

We have already indicated the methods adopted for eliminating the arsenious acid derived from pyrites.

It is not advisable to concentrate the acid beyond 140° to 150° Twaddell in ordinary leaden pans, for not only is the lead destroyed very rapidly both by the heat and corrosiveness of the stronger acid, but also it would be productive of great waste of fuel. When a greater degree of strength is required it is attained by boiling in retorts of glass or platinum. There are, however, some plans proposed by which

higher concentration may be prosecuted in leaden vessels and to these we shall have occasion to refer hereafter.

Glass Retorts.—It will be readily believed that the boiling of such a powerfully corrosive substance as sulphuric acid requiring a temperature three times as great as water in extremely thin glass flasks holding some 3 cwt. each, is an operation of the greatest delicacy, not to say danger; and it is therefore of primary importance that the “Retort house” shall be built of brick or concrete with a most durable roof, and that every crevice shall be securely stopped against the admission of the slightest draught, or drop of fluid, whether rain or acid. The house should also be roomy enough and especially of a good height, for as ventilation is inadmissible, sufficient space must be left overhead for the accomodation of the extremely pungent and irritating fumes which pour off the retorts during the time that they are being drawn off.

Fig. 53 shows in plan, elevation, and section, the arrangement and construction of the furnace and the iron pot which holds the glass retort.

A is a cast-iron pot, weighing about 4 cwt.

a furnace bars.

b bed plate.

c door frame.

d flue holes, one on each side of the fireplace.

e flue holes leading into the main flue *f*. These holes should not be all of the same size, but should increase in proportion as they are farther removed from *g*, so that the amount of draught may be equally distributed among the furnaces. The one nearest to *g* may be 3 in. $\times$ 3 in., the one farthest from it 3 in. $\times$ 6 in., and the others of intermediate sizes.

f flue leading to

g flue hole, about 12 in. $\times$ 9 in., fitted with a damper and leading to chimney. In the figure it is supposed to be at the end of 6 retort-furnaces, that being the number of

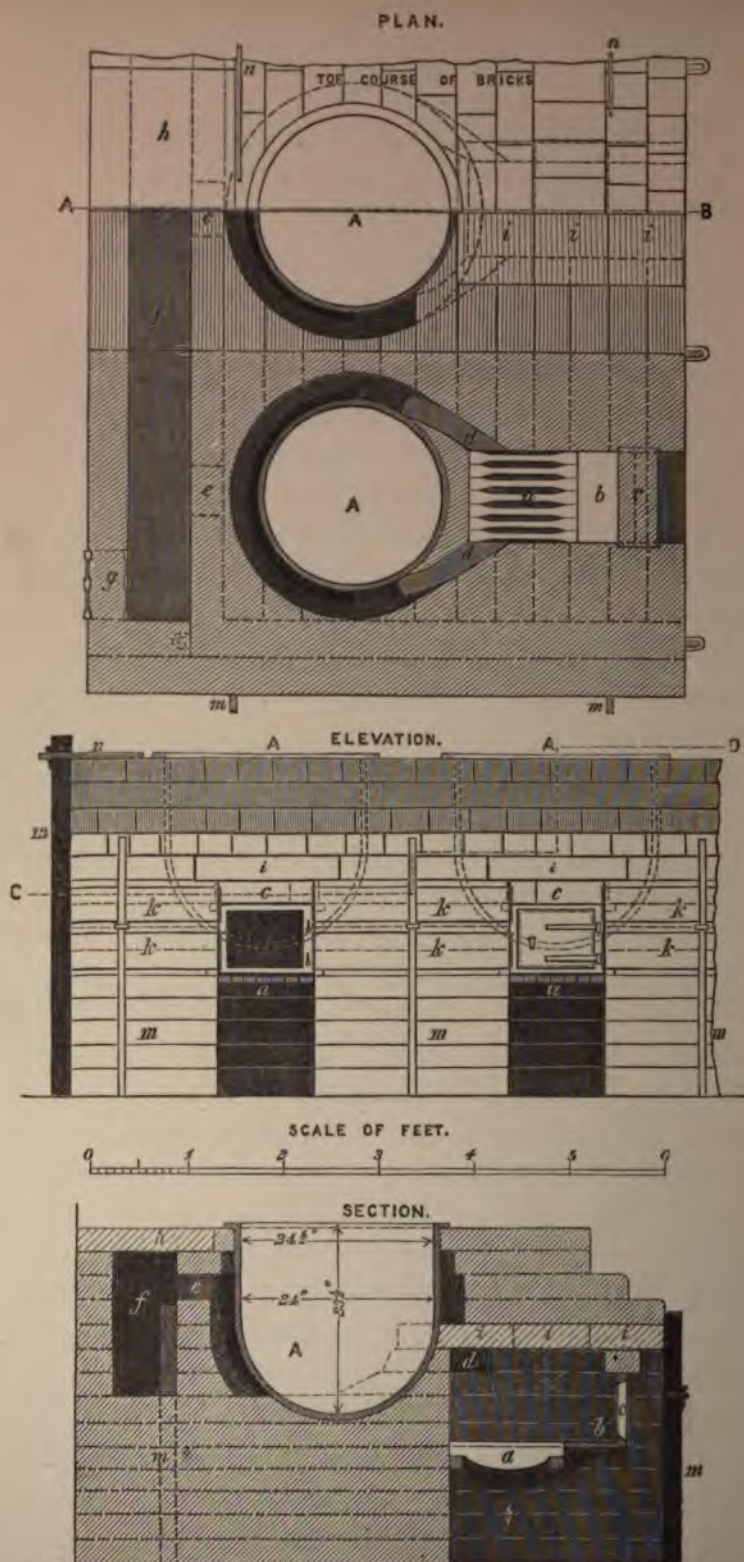


FIG. 53.

retorts suitable for a pan as shown in figure 51, but with 12 retorts it may be placed in the middle.

h fire lumps covering in the flue *f*.

i fire lumps covering the furnace fire.

k thin plates of iron to strengthen the front brick-work.

m perpendicular tie bars.

n horizontal tie rods.

In fig. 54 are seen the furnace doors and door frames and the fire bars. The furnace doors weigh $13\frac{1}{2}$ lbs.; the door frames, 26 lbs.; and the fire bars, $10\frac{3}{4}$ lbs. each. The furnace bed plate *b* (fig. 53) measures 18 in. $\times$ $6\frac{1}{8}$ in. $\times$ $\frac{5}{8}$ in., and weighs $19\frac{1}{2}$ lbs. The fire bar bearers measure 18 in. $\times$ $1\frac{3}{4}$ in. square, and weigh 14 lbs. each. The cast-iron tie bars, *m*, measure $2\frac{1}{4}$ in. $\times$ 1 in., those at front and back of furnaces being 2 ft. 8 in. long, and weighing 20 lbs. each. The wrought-iron tie rods connecting them are of $\frac{7}{8}$ in. square iron. The tie bars shown at each end of the row of furnaces are connected by tie rods, *n*, of round iron, $\frac{5}{8}$ in. diameter, but these would not be necessary if the furnaces were enclosed between strong retaining walls at each end.

Choosing Retorts.—The glass retorts may be about 3 ft. in extreme length and nearly 2 ft. in diameter, or of any smaller size desired. They should be ordered of such dimensions as will suit the iron pots. If the pots be of the proportions shown in fig. 53, the retorts should be 36 in. deep and $22\frac{1}{2}$ in., or, at the utmost 23 in., diameter. With any greater diameter there will be danger of their sides touching the pot should they shift ever so little from the centre, and if much smaller, they will not contain so much acid, and it will require a stronger fire to make the acid boil, owing to the greater space between the sides of the pots and the sides of the retorts. The inside diameter of the neck of the retort should be about $3\frac{3}{4}$ in.; if it be much smaller, the steam arising from the boiling acid will be

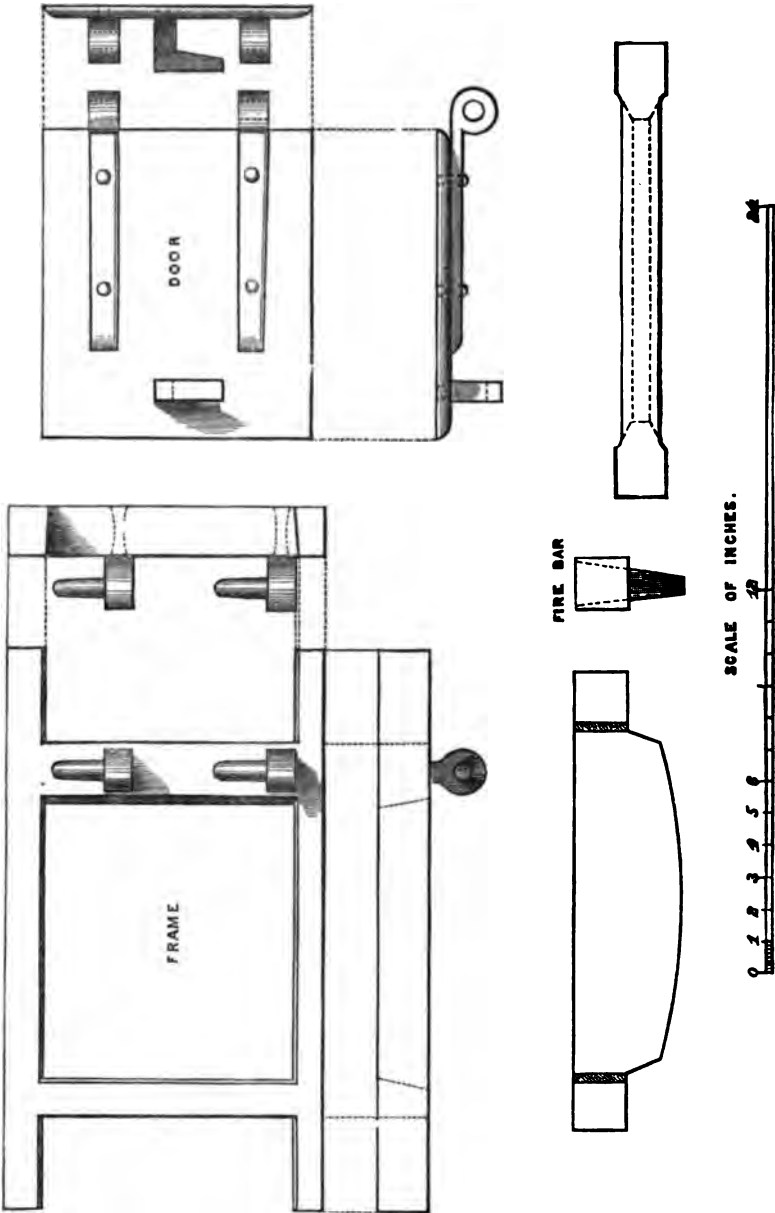


FIG. 54.

impeded in its exit, and the concentration of the acid will be retarded. The bottom and sides of the retorts are extremely thin, the bottom being about $\frac{1}{32}$ in. and the sides less than $\frac{1}{30}$ in. thick. The rim is rather more than $\frac{1}{4}$ in. thick. A retort of the above proportions will weigh about 18 lbs. The diameter of a retort is best ascertained by passing a tape measure round its largest part, which should be near the middle, and dividing the result (its circumference) by 3.1416. Thus, if the circumference were 6 ft., then $72 \text{ in.} \div 3.1416 = 22.9 \text{ in.}$ diameter.

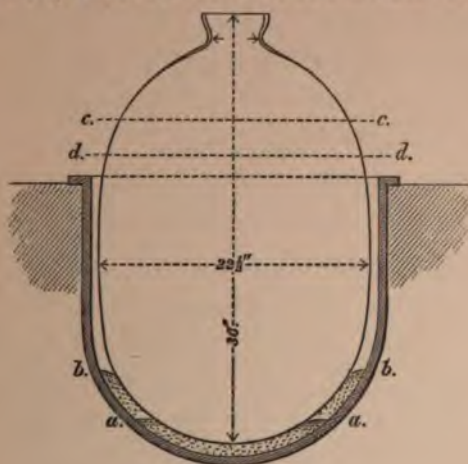


FIG. 55.

Setting Retorts.—

Before commencing to “set” the retorts, it must first be ascertained that the pots are absolutely dry. Some sand must then be thoroughly dessicated and afterwards sifted through fine wire gauze to free it from any small gravel, &c., that may be in

it. Put some of this dried sand into the pot and spread it over the bottom with your hand, giving it the hollow shape of the bottom of the pot until the layer of sand is of a uniform thickness of about 1 or $1\frac{1}{4}$ in., and reaches as far up the pot all round as shown at *a a* in fig. 55. Take the retort by its neck with both hands, and lower it gently into the pot till it rests on the bed of sand. Place it as nearly as possible in the centre of the pot, and take care that it stands perpendicularly. Should it be found that the sand does not support the retort (when left alone) in an upright position, a little sand must be poured in between the retort and the pot on whichever side support is wanting. When satisfied that the retort is well placed in the pot, take more sand and

pour in all around the glass till it rises as high as *b b*. The retort will then be ready for filling with acid.

Filling Retorts.—A leaden pipe, $1\frac{1}{2}$ -in. bore, leads from the evaporating pan to the retorts (see *q*, fig. 52), lying on the brickwork in front of the retorts. The end of this pipe is closed, and opposite each retort a very stout leaden pipe of $\frac{1}{2}$ -in. bore is fixed into the large feed-pipe in a perpendicular position. These small pipes are long enough for their mouths to be above the level of the top of the pan, when supported upright. When the retorts are to be filled the small pipes are bent down, so that their ends enter the mouths of the former, as shown in fig. 56. The pan syphon, already explained and figured on p. 135 is then set, and the acid flows along the large feed-pipe and up through the small pipes into the retorts. A little before the desired level of acid has been obtained in the retorts, the syphon cup is raised, and the supply thus cut off, the small pipes are allowed to drain themselves, and when they have quite finished running they are bent back carefully into their perpendicular position, and kept in place by their bent ends being hooked into loops made on the ends of stout copper wires suspended from the ceiling, see fig. 56.

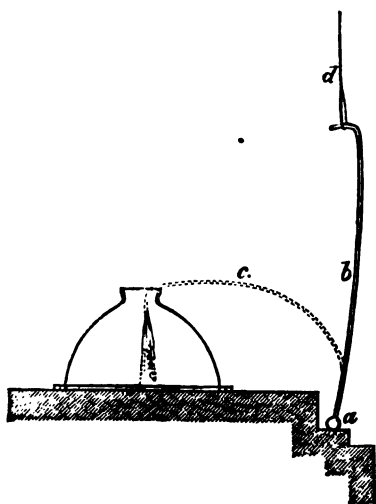


FIG. 56.

a is the large feed-pipe in section.

b one of the small pipes held up when not in use.

c ditto in the act of filling a retort.

d copper wire.

Fill the retorts as high as the line *cc* (fig. 55) if the acid be quite hot, but only to the line *dd* if cold, as it will rise from *d* to *c* in getting hot.

When the retorts are newly set, or if, through not being worked for some time, they have become cold, they must be filled with cold, or nearly cold, acid, or they would break. After they have been worked for a day, the concentrated oil of vitriol which is in them, and which is still very hot, is drawn out by syphons at say 6 a.m. on the following morning, and they are then *at once* refilled with *hot* acid, which has been boiled in the evaporating pan to the proper strength of say 150° Twaddell, during the preceding day and night. Cold acid must never be put into hot retorts, nor hot acid into cold ones.

The acid, whether hot or cold, should be about 150° Twaddell. If much weaker, it would boil so violently in the retorts as to be dangerous, and so much concentrated acid could not be produced from it; on the other hand, if made much stronger in the pan, its action on the lead would be very destructive.

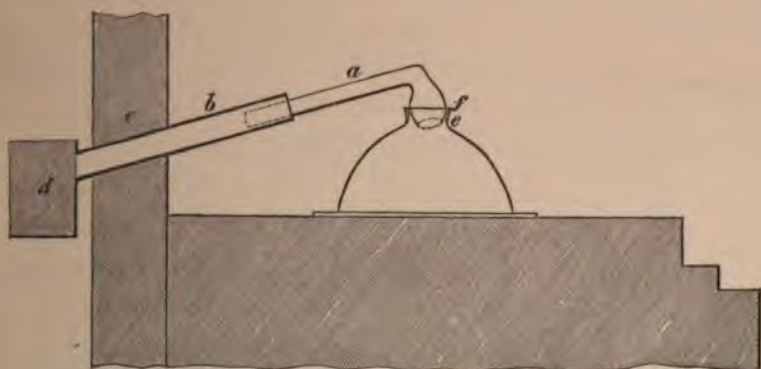


FIG. 57.

Retort Arms.—As soon as each retort is filled, and the pipe has been put up into its place, the glass arm (see fig. 57) should be put on. These arms (one to each retort) connect the retorts with leaden pipes passing through the wall of the retort house into a leaden trunk, leading to a small coke condenser, which receives and partially condenses the steam which comes off the acid during ebullition. The

arm consists of a stem and a bulb. The stems are inserted say 5 or 6 inches into the leaden pipes, which project a sufficient distance from the wall, and the bulbs are placed in the mouths of the retorts.

a retort arm.

b leaden pipe.

c wall of retort house.

d trunk leading to condenser.

As the bulbs of the arms, also the mouths and necks of the retorts vary in size, each retort has its own proper arm, which fits it. The leaden pipe *b* must be large enough for the stem of the arm to fit it loosely, as it must not be bent or moved when the arm is put on or taken off the retort. The bulb of the arm must be large enough to rest on the neck, *e*, of the retort, and not pass through it, but must be small enough to enter the mouth, *f*, of the retort, with a little room to spare. A small strip of (6 or 7 lbs. to the square foot) sheet lead about $\frac{1}{2}$ in. wide, and 6 or 7 in. long, should be bent and hung over the edge of the mouth of each retort, to admit of a little ventilation. This strip may be placed at any point on the mouth, choosing that spot where it will best support the arm in its proper position.

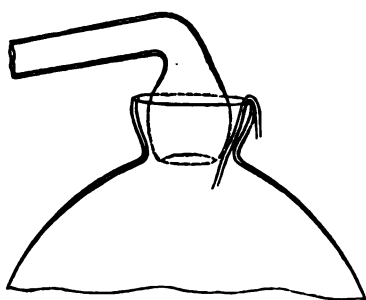


FIG. 58.

In fig. 58 it is placed at the front of the retort mouth, but the best place is generally somewhere on one side or the other, and not at the front nor back.

Working Retorts.—While the retorts are being filled, but not before, their furnace fires may be lit, quickly done

by putting into each furnace a shovelful of fire from the furnace of the evaporating pan. Moderately good fires should be made and maintained until the acid begins to boil, when, for about an hour, very low fires will be suffi-

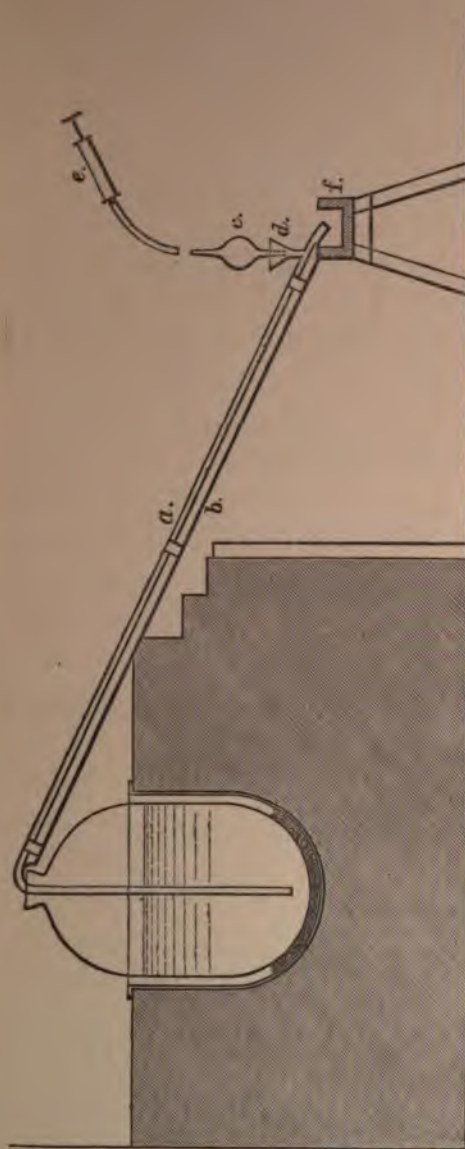


Fig. 89.

cient; but, as the acid continues to boil and get stronger, the fires must be pushed. In fact the firing must be regulated entirely by the boiling, which should never be allowed to cease, but should be gentle during the first hour or so, to guard against the acid suddenly leaping over and causing a destructive and it may be serious and even fatal accident. The mode of firing the furnaces is as follows:—Commencing at one end of the row, open the door of No. 1, stir the fire, and take out any clinkers, shut the door, and proceed to do the same to Nos. 2, 3, &c., in rotation to the end; then recom-

mencing at No. 1, put a small shovelful of coal on each fire. Sufficient coal for the day's wants previously broken to a convenient size, must be always brought in during the time of filling the retorts in the morning, so that no door needs to be opened during the process of the boiling. Hot acid run into the retorts will commence boiling in from 3 to 4 hours, and must be kept boiling for 6 or 7 hours.

The moment each retort *commences* to boil (as soon as a few bubbles rise through the acid, which must be anxiously watched for) the fire must be checked, by opening the furnace door for 5 or 10 minutes, or, if the boiling should increase rapidly, it may be necessary to cool the furnace all possible by taking out the fire and putting in wet ashes instead, which should be kept at hand in case of need. The weaker the acid is when put into the retorts, the more watchfulness must be exercised during the first stage of the boiling.

Emptying Retorts.—Early on the morning succeeding the day of boiling, the acid (now become concentrated) may be drawn off by a syphon into a cooler, where it will remain until its temperature will admit of its being put into glass carboys say at 100° Fahrenheit. The syphon is shown in fig. 59 placed in the retort ready for setting.

a is the syphon formed of a piece of $\frac{1}{2}$ in. bore leaden pipe, bound to

b a small piece of wood.

c a glass globe with two tapering tubes, the end of one tube being inserted into

d a lead funnel, in which *c* is hermetically sealed by a mixture of melted brimstone thickened with a little sand.

e an exhausting syringe, to the mouth of which is attached a short piece of India-rubber tubing.

To set the syphon, one person takes a small piece of sheet India-rubber, say $\frac{1}{8}$ inch thick, and holds it tightly against the mouth of the syphon, to stop the passage of air, whilst a second person takes the syringe and slips the end of its flexible tubing over the end of the upper tube of the glass globe. Then working the syringe a few strokes, the air becomes exhausted from the syphon, causing the acid to flow through it, and commence to fill the glass globe. The syringe is then removed, and the piece of India-

rubber is quickly withdrawn from the mouth of the syphon, the acid continuing to flow until the retort is nearly empty.

f a wooden trough lined with lead (shown in section), to catch the acid from the syphon and lead it to the cooler.

The syphons do not reach quite to the bottoms of the retorts, so that about a gallon of acid is left in each. This makes the refilling safer, as, although the retorts are very hot, the acid with which they are refilled may be much hotter; the difference of temperature is lessened by the acid left in them.

Precautions, and Cleaning Retorts.—Should any acid accidentally be spilt over a retort and reach the sand on which it stands, or should the sand become wet from any cause, it would be unsafe to work the retort. It must be taken out of its pot, the sand redried and sifted, and the retort put back again, as before described. The retorts, when empty, should be pressed sideways at their necks, occasionally, to move them slightly, to be assured that no damping or wetting of the retort sand has escaped observation. Should this have been the case, the sand on drying will have fixed them firmly to the pots, and it will be found that more pressure is required to move them than is ordinarily the case. Under these circumstances they should on no account be worked, but be lifted carefully out of the pots, and the sand removed, &c. The interior of the retorts should be inspected from time to time. Sulphate of lead and other impurities will gradually accumulate at the bottom, and, if allowed to remain too long undisturbed, will become hard and very difficult of removal. To clean a retort it is taken out of its pot, laid on a piece of sacking held off the ground by two men, and rocked about with a little concentrated acid in it, which by degrees will wash off the sediment. The acid will be renewed as often as may be necessary. When a retort is taken out of its pot

it should never be put in again without taking out all or the greater part of the sand, and preparing the bed as already described.



FIG. 60.

The basket shown in fig. 60 is handy for holding a retort. The upright rods and top ring should be of white rods, the remaining parts of brown rods, and the bottom of the basket should be flat. It should be more than half filled with soft hay before receiving the retort.

Violent Ebullition. — To prevent jumping ebullition

in the retorts, it may be worth while to try the effect of a small piece of platinum sponge put into the retort. Scarcely any other porous body would remain unaffected by the acid. Richardson and Watts have recommended the use of sand or sulphate of lime for this purpose, but we cannot approve of either of these substances, on account of their liability to form a solid cake on the bottom of the retort, which would be productive of far worse results than the violent boiling which they are intended to check.

Robierre puts 12 grammes of thin platinum plates into 320 cubic centimetres of sulphuric acid, and says that the ebullition is then as gentle and regular as can be wished.

Mons. Raoult recommends the use of fragments of gas carbon for the prevention of bumping during the distillation of sulphuric acid. He states that the ebullition then proceeds with perfect regularity, while the carbon is practically unacted upon. Some pieces weighing altogether 4.567 grammes, after having been subjected to the action of boiling acid for 8 hours, weighed 4.501 grammes; they had therefore lost only 0.066 gramme, and had generated but a

trace of sulphurous acid. It is said that after this treatment the gas carbon produces marks on paper similar to those made by plumbago.

Coolers.—After concentration in either leaden pans to the strength of brown oil of vitriol, or in glass retorts to rectified or white oil of vitriol, the acid must be cooled down to about 100° Fahr., before it will be safe to put it into carboys for removal. This is conveniently done in shallow vessels made of thick sheet lead, fitted into suitable cases of wood or iron, sufficiently strong to bear the weight of the acid and lead. The lead sheet must be turned over the edge of the box which it lines, to avert the possibility of some drops of acid occasionally finding their way down between the two materials, and causing trouble and annoyance by destroying the outer case of wood or iron. Iron is preferable to wood for the outer casing, as it enables the acid to cool much more rapidly. It is well to raise the cooler upon low brick walls or other suitable support, so as to admit of the easy passage of cool air underneath, but it must not be raised so high as to inconvenience the ladling out of the cool acid into carboys. The cooling room should not have any means of access from the retort house, the acid being run into the cooler by means of a trough, as described, passing through the wall. The room should be kept scrupulously clean, to prevent dirt getting into the cooler and spoiling the colour of the acid; it should also be well ventilated. In a works where steam power is used, a small fan may be driven at very trifling cost, and will greatly hasten the cooling of the acid. The cooler should be provided with a well for convenience in dipping up the acid. The number of coolers employed will naturally be regulated by the quantity of acid, and it should be remembered that a small bulk cools more quickly than a larger.

Chance's Patent Retorts.—In 1871 Mr. Henry Chance patented a plan by which he proposes to do away with the intermittent character of the concentration in glass retorts

is already described and to render the process continuous. The pyro acid of about 1.45° Twaddell, entering at one end of the apparatus, being concentrated as it passes through it, and arriving at the other end in a concentrated state. Mr. Chance's first idea was to make one fire do for, say 4 retorts, preferably of glass, placed in the same inclined flue, the fire being made under the lowest retort. The acid being caused to run from one retort to another in a direction opposite from that of the flames, &c., from the furnace, thus arrives at last in the hottest retort, where the concentration is finished. He considers, however, that in working on a large scale it would be preferable to have two series of retorts in combination (side by side), and to provide each retort with an independent furnace.

The fire from the furnaces passes into an air chamber, and from thence to a flue, which is common to the furnaces of one series of retorts. The retorts are supported at different levels in the hot air chambers in pans or dishes of iron, containing sand, in which the bottoms of the retorts are imbedded. The top of the hot air chamber is closed by rings. In connexion with the hot air chamber is a flue, into which, in case of breaking of one of the retorts, the acid passes, and is conveyed therefrom by a tube into a receptacle. The flame and heated air are prevented from striking directly against the sides of the retorts by means of deflectors between the furnaces and the hot air chambers. There are funnels by which the acid is conducted to the bottoms of the retorts, and tubules by which the acid overflows from a retort into that next below it, or finally passes out of the apparatus. These tubules may consist of a short inclined pipe on the shoulder of each retort, the said pipe delivering the liquid from it into a connector which conveys it to the funnel of the next retort. Each of the open mouths or tops of the retorts is fitted with a pipe or connector somewhat resembling the head of an alembic. These pipes open into a leaden flue, which is connected with the

chimney-stalk of the furnaces. The steam together with a little vapour of sulphuric acid escaping from the retorts passes into the flue, where the acid is condensed and the steam passes away. The condensed acid runs down an inclined leaden flue, and escapes by a pipe into a receptacle. The dilute acid to be concentrated is contained in a reservoir, from which it passes by a syphon into a vessel, a pipe from which delivers it into the top retort. The bottom retort delivers the concentrated acid into a vessel, from which it passes along a pipe of small diameter, into another vessel, and by a syphon into the delivery vessel, a pipe in the bottom of which conveys the acid into carboys or into other receptacles. The vessels and the connecting pipe are immersed in a trough of water, by which the acid is cooled: cold water is passed through the trough, and escapes therefrom at its top. The parts in which the acid cooled are made of lead, but the temperature of the acid as it escapes from the last retort is such that if it came immediately in contact with the lead, it would act thereon. To prevent this, a small cup of platinum is fixed in the vessel, upon which cup the stream of hot concentrated acid strikes.

In working the apparatus for the concentration of sulphuric acid, the reservoir is supplied with dilute acid having a specific gravity of 1.740 or 61° of Beaumé's hydrometer. As the acid passes through the retorts, it is necessary to keep it boiling very gently, or the flow of liquid from one retort to another will be made irregular. The heat of the several furnaces requires to be so managed that the acid shall be exposed to a higher temperature in the retort into which it is running than it was exposed to in the retort which it is leaving, but any one accustomed to the ordinary method of concentrating sulphuric acid, will have no difficulty, it is said, in managing the fires of this apparatus so as to produce the required gentle ebullition in the several retorts. Care must be taken that the stream

of acid delivered into the top retort is somewhat less than the several tubules are capable of conveying.

The process of concentration as conducted in these retorts is continuous, and may be carried on for several weeks without interruption. At intervals of about a month it is preferred to suspend the operation of the apparatus, for the purpose of cleaning out the flues, and making necessary repairs.

The patentee says that by constructing and arranging retorts or apparatus for the concentration of sulphuric acid and other liquids in the manner described, great saving in manual labour, fuel, and plant is secured.

Platinum Retorts.—The adaptation of platinum in lieu of glass for the construction of vessels for the concentration of sulphuric acid, possesses so many advantages and has been brought to such perfection by Messrs. Johnson, Matthey, and Co., of Hatton Garden, that it is fast displacing the older plan in all large works.

We are indebted to the kindness of Mr. Sellon, of that firm, for the following figures and particulars of their valuable apparatus, including the most recent and improved modifications introduced by them.

Original Plan.—Originally these retorts or “boilers,” as they are called, were made of very large dimensions, even capable of holding 200 gallons of acid at one time, and the various pieces of platinum of which the boiler was formed, were joined together by lapping the edges, and fastening them with gold solder. In some out-of-the-way countries these old boilers may still be found, having several pounds weight of gold about them. Indeed, foreign manufacturers still adhere to the expensive and inadequate system of soldering the joints with gold.

This old-fashioned form of boiler possessed some advantages over glass retorts in reducing the cost of labour and fuel, and in rapidity of concentration, but it left much to be desired. Especially the prime cost was very great on

account of the weight of metal, and the intermittent nature of the process caused great wear and tear of plant, as the non-homogeneous joints suffered severely from the contraction and expansion of the metals consequent upon the alternate lighting up and letting out of the fire.

Since 1860, the autogenous process of soldering, which has already been described, has been applied to platinum as well as to lead, and sulphuric acid makers have learnt to regulate the inflow and outflow of the acid, so as to make the boiling process continuous instead of intermittent.

First continuous Form.—Figure 61 shows an early form of retort for continuous concentration, which followed the simple pans or dishes of more or less depth, either with leaden covers or worked under large leaden hoods or cupboards. It was used with a series of leaden concentrating pans, and resembled a huge kettle, with a diameter varying from 12 to 42 inches.

A retort or boiler.

B arm for carrying off the vapours generated during concentration.

CC cooling syphon.

a acid feeder to retort.

c air-hole for setting syphon.

d charging hole for setting syphon.

e feed pipe of cold water to cooling syphon.

f exit pipe of heated water from cooling syphon.

g exit tap of cooled acid from cooling syphon, by which it is run into carboys.

The ebullition was rendered violent in this form by the acid entering in too sudden a manner. The cooling syphon was also found to be inconvenient, both from its unwieldy length, sometimes 20 ft., and from its giving great trouble in cleaning, when using impure acid.

The next improvements consisted in reducing the depth of the retort (thus economizing metal), and in delivering the pan-acid on to a perforated ring inside the retort, as

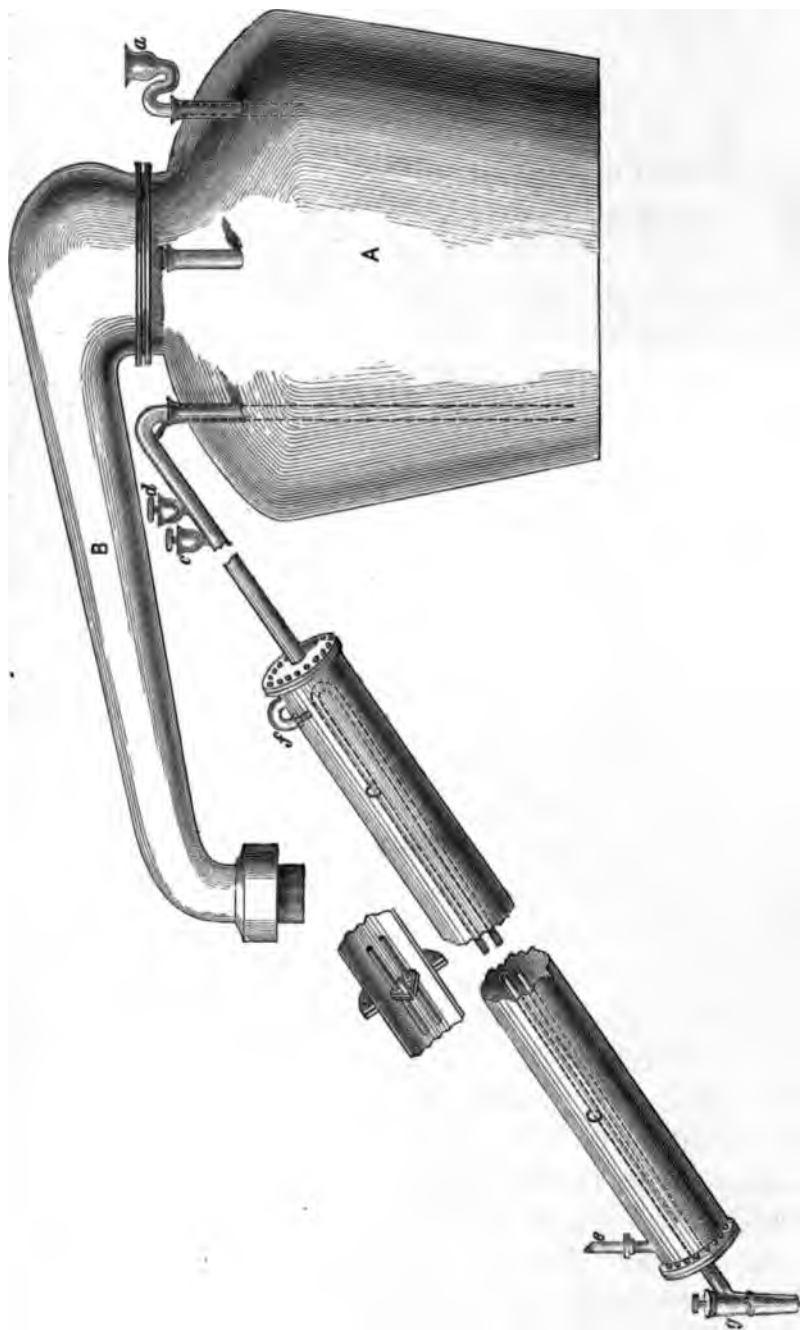


FIG. 61.

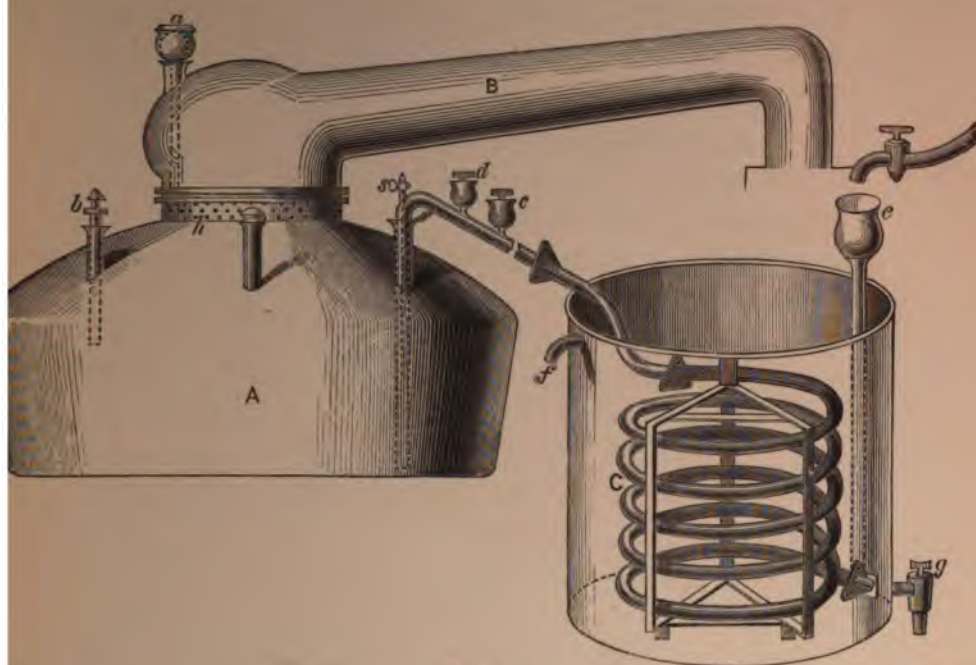


FIG. 62.

shown at *h* (fig. 62), in a gentle stream, so that it should trickle through the holes and down the *sides* of the retort, on to the *surface* of the acid already in the retort; also in making a raised in place of a depressed arm, it being found by experience that during the violent boiling which takes places at times within the still, strong acid was mechanically carried over with the distilling vapour, and thus lost. A further improvement also was made in an air tap, *s*, so arranged that the syphon would discharge itself if, by carelessness or accident, the supply of acid to the boiler or still should cease, thus avoiding the chance of damage by burning the sides. As the concentration progresses the acid increases in density, and therefore sinks by its own weight to the bottom of the retort, whence it is drawn by the cooling syphon. The flow into and out of the retort can be regulated by the taps feeding into *a* and at *g*.

The "coil"-syphon is also an improvement on the older form, economizing space and giving greater cooling surface.

Mr. Petrie's Improvements.—One of the earliest material improvements in the form and construction of platinum retorts for sulphuric acid concentration, was exhibited by the firm of Messrs. Johnson, Matthey, and Co., in the Paris Exhibition, 1855, being a vessel having great extension of bottom surface for exposure to the fire, and for working with a shallower layer of acid than with the deep form of still then in general use.

This vessel, which was lined with gold, was made after the designs of Mr. William Petrie, a gentleman who had devoted much scientific attention to the subject, and who, later on, improved this form into that exhibited by the same firm of manufacturers at the London Exhibition, 1862. The improved shape and proportion embodied, amongst many others, the following advantageous points:—

1. The shallow form of apparatus.
2. The arrangement of the boiler on an iron stand, with the concave bottom exposed directly to the heat of the fire.
3. Two or three stills working together, one into the other.
4. Continuous inflow and outflow of acid.
5. System of exit by a tube passing from a compartment in the centre of the boiler, through the side to the refrigerator.
6. Cooling the acid by forcing it to run into a large cooler containing comparatively cold vitriol surrounded with water.

It has since formed the basis of some of the most recently adopted and approved principles, but did not find general favour at the time in consequence of the deep-rooted prejudice in favour of digesting a large body of acid, for which purpose the older form was a necessity.

The end in view in this form of platinum apparatus for concentrating, was especially to make its cost to be but a fraction of that which was ordinarily incurred for obtain-

ing a given rate of production. A vessel on this plan, including the head and other fittings, platinum supply tap in duplicate, platinum, lead and stoneware coolers, and stoneware store jar, and the necessary connecting pipes and apparatus as shown, cost about 650*l.*, the variation depending upon the exact weight of platinum used. It was capable of rectifying at the rate of more than 2 tons per diem, from 80 per cent. acid, but was offered as rectifying 2 tons per diem from acid at 154° Tw., which is the strength desirable for the best economy in working, having reference alike to cost and wear of the rectifying apparatus.

His Boiler or Still.—The sides and top of this are made of such general form as to cover the heating surfaces and to enclose the greatest space for the liquor and its boiling froth by the least surface of platinum. The upper parts of the vessel, not needing strength to support the liquor, are not made so thick as the rest.

As regards the heating surfaces, the form of the vessel and its setting are so arranged that a greater proportion than usual of the heat is transmitted through the bottom or otherwise by means of *direct radiation* from a surface of clear fire, as distinguished from heating by a draught through flues, in part out of sight of the fire, hence a smaller heating surface suffices, and the metal does not so readily become coated with non-conducting fur or dust, as in the usual flues. Those surfaces of the platinum which are to heat the liquor are made dead or rough externally.

For the lower parts of the boiler, two alternative forms are adopted, according to the particular service for which it is destined. Where circumstances require the shallow form of vessel, it has the advantages of affording a more ready evolution of the steam or watery part of the acid, and any variations of height of the liquor do not endanger the contact of fire with parts not reached by the liquor within, and it allows the bottom to be made of considerable width,

without the usual supports or obstructions to the direct heat of the fire ; it also prevents much hydrostatic pressure, which otherwise necessitates greater weight of metal and cost, and aggravates the effects of leakage, should it occur.

The mode of setting this vessel is extremely simple and safe, being on an iron ring, which is, in most cases, formed so as to receive heat and serve as a heater to the lower circumferential part. In this way the few tiles placed around the boiler, higher up, are all that need to be removed in order to take out the boiler for cleaning or repairs. This is a consideration, in view of the occasional fouling of the interior. The furnace too, thus arranged, is more simple than usual, its flues passing from the boiler to leaden pans, and where these are not already erected on the ordinary plan, the construction recommended for this bank is such as to reduce several of the current expenses, especially in fuel.

The head and beak are modified from the old pattern, so as to attain the utmost economy of precious metal and yet to give more free vent to the vapour.

The vessel is intended to work with a continuous inflow and outflow of liquor, regulated by a single tap, namely, one to supply the inflow. Even when two or three boilers are used together, only one such tap needs to be regulated. The outflow is effected by a self-acting process of overflow, which prevents the inconveniences of the usual syphon, such as the risk of inattention to its tap allowing the liquor to run itself too low, or to rise too high ; also the appliances and trouble connected with keeping it charged, whereas on the improved plan there is no outflow tap, or in cases where it is made it does not need any attention, but is set, once for all, more than sufficiently open, and is closed only on emergency. The overflow is by a nozzle into a pipe, connected by a spherical slip-joint, a plan which has proved to be remarkably convenient, safe, and dry. The overflow nozzle is chiefly supplied, when the boiler is in full work, by

one of two plans, determined by circumstances. On one plan the outflow is by a trapped pipe from a "collecting vessel" fixed inside the boiler, the size and height of the collecting vessel being such that in regular working it receives the rising froth or bubbles so freely that the froth does not rise and boil over. It supplies to the outflow pipe only such liquor as has discharged its commingled vapour, while thus settling down in this vessel where no boiling takes place. This arrangement is considered important as securing a free outflow, not restrained by commingled vapour during rapid working, and as tending to realize a certain fixed level for the liquor, the boiling of vitriol being an action difficult of control.

The Cooler.—The process of cooling the vitriol as it flows from the boiler to the storing jar, is performed at the first stage of the process, partially, if not completely, in a suitably constructed vessel of platinum. The end of the outflow pipe drops into a central tube in the platinum cooler made to so dispose of the hottest liquor as to prevent fumes from rising. This cooler performs the part of the usual long platinum tubing of a sleeved syphon, or of a worm, &c., but it is neither so costly, so liable to injury, so troublesome to fix and remove, nor to keep always charged and in working order; neither does it need to be disturbed at all, when the boiler has to be removed and replaced. Furthermore, it is much more easily inspected and cleaned than the sleeved syphon when the gradual accretion of furry matter, internally and externally, may render that step necessary. The economy of water for this cooler cannot be excelled, as it acts differentially, by a regular gradation of temperature, the water heating upwards while the vitriol cools downwards.

The Indicator or Gauge for Gravity.—This instrument is inserted in a tube in the top of the boiler, and by it the rectifying is regulated with unusual accuracy and ease. It acts automatically and continuously and has a dial traversed

by an index showing, at any minute, the strength of the boiling acid without the trouble and delay of obtaining a sample from the outflow, cooling it and trying it by a hydrometer. It shows, too, a much smaller fraction of the last degree of gravity than the hydrometer itself can show.

The indicator acts by the variations of heat at which each different strength of acid boils ; it is a novel application of the spiral coil, already known, of a compound lamina of two metals of unequal expansion by heat, here arranged with important improvements, and is said to serve also as a superior pyrometer for general purposes.

Mode of Setting.—Fig. 63, partly in section, shows the mode of setting when three such boilers are used connectedly. When one only is used, its setting, and the furnaces and cooler connected with it, will be represented by the lowest of the three here shown.

AA the furnaces in section. The dotted lines just below the boiler indicate blunt edges or arrises, in an internal and external sense, formed by the junction of the square bottom with the circular top of the furnace.

BB the boilers, set in their circular cast-iron seats, seen externally.

C the pipe, end view, leading to the worm or other condensing apparatus for the fumes evolved from the beaks of the boilers.

D the conduit syphon, a fixture fitted with the improved platinum tap, to supply the liquor continuously (as regulated) either from the improved form of pan (partly shown) or from ordinary leaden pans, to the highest boiler, whence the acid passes by the connecting pipes through the other boilers, and flows out continuously into any cooling apparatus, but preferably into

E the improved platinum cooler. When more than one boiler is used this cooler is connected with other coolers, having their central vessels made of stoneware instead of

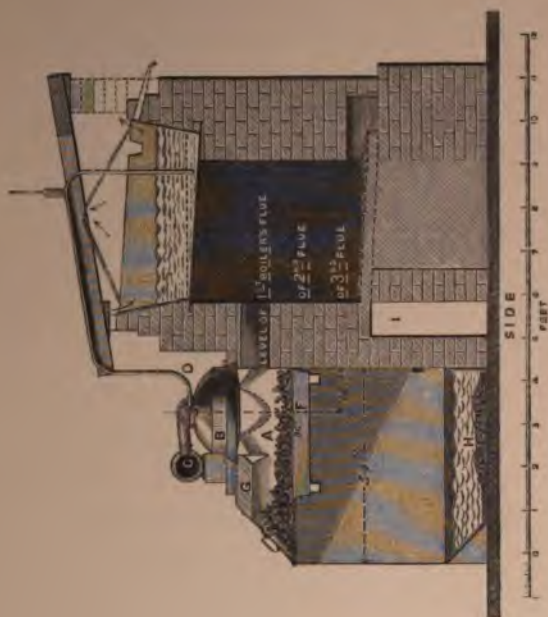


FIG. 43.



M

platinum—in the background not here shown. The flow both of the water and of the acid is consecutive, in opposite directions through the whole set. Thence the rectified acid flows into a 30 or 40 gallon stoneware jar furnished with a stoneware tap for drawing it off at convenient times into carboys.

FF the fire bars, deep and narrow. They are preferably 5" deep, by 1" wide at the upper side, $\frac{1}{2}$ " wide at the lower side and with $\frac{3}{4}$ " spaces.

G the arch of the furnace. Most of the joints of its brickwork are left "dry" without mortar or clay, except at the front or coolest end of it, the object being to admit numerous jets of fresh air, flattened very thin and strongly heated by passing through the brickwork, to play on the upper surface of the gases arising from the coal, to increase the heat under the boiler, while reducing the temperature of the arch, and changing most of the smoke into flame. Welsh coal is preferable. Each charge of coal is moved three times before it reaches the farther end of the furnace, namely, before each of the two next charges is inserted, so that the farther end of the furnace is always burning clear without flame, the middle partly so, and at the front is black coal, and these gradations should not be intermixed when each portion is stirred and pushed farther in on the addition of a new charge.

HH pans for water in the ash-pit.

I main ventilating flue leading to

JJ branches from it between the furnaces, to prevent their sides from "sweating" by excessive accumulation of heat.

KK the places for the Indicators in the boilers, to show continually the actual degree of concentration of the acid, without taking out a sample and cooling it.

There will necessarily be a flue to lead the waste heat from under the boiler to beneath the pans, and in this way as great an economy of fuel is effected as when much more

platinum is employed at proportionate cost to admit of a flue being led around the boiler, so as to absorb a greater relative proportion of the heat into the platinum from less intensely heated surfaces, and therefore with less effect per superficial foot of platinum employed. This boiler is not intended to be set without some such arrangement for utilizing the heat which passes from its furnace.

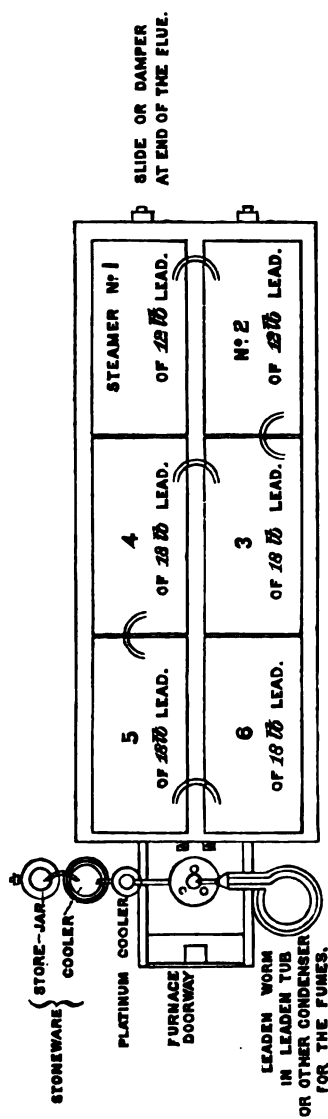


FIG 64.

The flue, on leaving the furnace, divides into two under the pans; this enables the manufacturer to shut off the draught which comes from the furnace, into either flue, by inserting a brick tile or stone endwise through one of the two holes *AA* (fig. 64) from above, between the boiler and the pans. In this way, when any pan leaks, the flue under it can be thrown out of use, without stopping the working of the boiler and the pans on the other side, while the leaking pan is left to cool, and is then emptied and repaired. During such an event, the acid is led successively through the pans (Nos. 1, 4, 5, or Nos. 2, 3, 6) on one side only of the bank. But when all the bank is in regular working order, the fire passes into both flues, and the acid is led alternately by means of hand syphons from side to side of the bank, thus passing successively

through pans Nos. 1, 2, 3, 4, 5, 6, and thence to the boiler.

The pans rest on iron plates slightly inclined (at 1 in 20) from the middle line of the bank towards each side, so that any leakage is thrown outwards instead of accumulating and sinking into the brickwork. The lower sides of the pans have about 7 in. width of their bottom resting on the wall, and not over the flue; this is to afford a lower corner, not heated, to which dirt and sediment can be raked from the other parts of the bottom, which is thereby prevented from overheating and too rapid destruction of the bottom of the pan. Their sides incline outwards slightly (1 in 20), which preserves their shape better.

The syphon of $\frac{1}{2}$ " or $\frac{3}{4}$ " leaden pipe leading from the last pan to the boiler is provided at one end with a platinum tap, while the other end, which stands in the pan, is bent upwards with a short curve, so that the syphon shall not lose its charge when lifted out of the liquor, provided, of course, that the platinum tap be first shut off; by this arrangement the syphon can be charged at leisure, when out of the pan, with cold vitriol, and then placed quietly into its position in the pan without danger of losing its charge.

Two such syphons with platinum taps ought always to be kept in position, side by side, one in use, the other with the platinum tap shut off, but ready to be turned on at any moment to supply the inflow funnel of the boiler, without trouble, whenever that syphon which is in work fails in its duty, either by a bit of dirt getting into it and obstructing the flow, or by losing its charge through any accident, thus the working of the boiler will not be interrupted, while the disordered syphon can be put to rights without hurry.

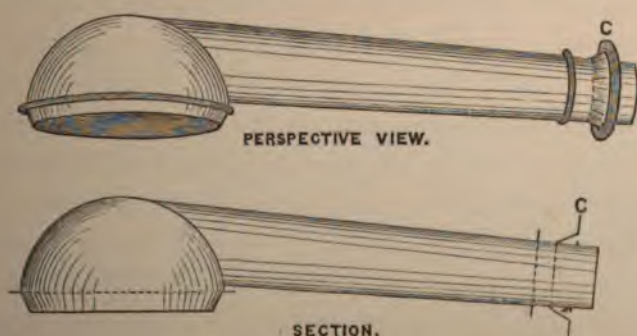
The platinum tap is attached very simply and effectively to the leaden pipe by pressure around the outside of the latter, applied by an iron screw compress, and when once fixed, the tap will remain long without needing to be com-

pressed again. There is a strong toothed ring of platinum forming part of the inner end of the tube or stem of the tap, inside of the leaden pipe, and the pressure beds the teeth and partly this ring into the internal surface of the leaden pipe. Each of the platinum taps supplied with the apparatus is thus attached already to a short piece of leaden pipe which can be united, by the plumber's blow-pipe, or in any other way preferred, to a proper length of pipe to form the syphon.

The two last pans are placed as close as possible to the boiler, in order that the syphon with the platinum tap may be short, and therefore easily handed about, so as to prevent difficulty whenever any obstruction should occur to its flow.

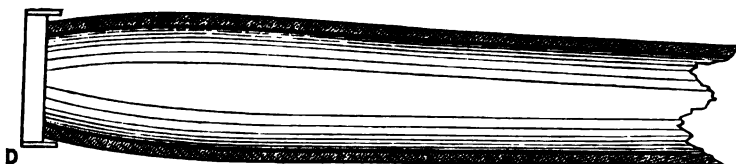
Conducting away the Gases.—The steam and acid fumes escaping from the head of the boiler may be taken into a condenser of any desirable form, or into the leaden chambers, but in any case it will be necessary to describe the manner in which the connexion shall be made between the platinum retort arm, and the condenser or conducting pipe.

Sometimes a glass adapter is used, or what answers well is one made of coarse earthenware made from clay, with as much coarse siliceous sand as can be possibly worked in with it, such adapter being connected by a simple slip-joint. But the plan provided for with this apparatus is to take the platinum beak, which projects from the head of the boiler, at an incline of 1 in. down in 12 horizontal, and slip the end of this beak into a collar of platinum, C, thus :—

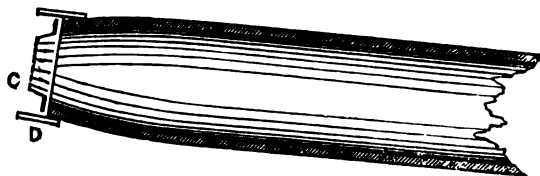


This collar C is to be first fixed to the end of a leaden pipe 4 in. internal diameter, and about $4\frac{1}{8}$ " external, having its end dressed in or contracted to $3\frac{1}{4}$ in. (see next figure), with its end cut quite flat, so that a flat disc will be in close contact all round it; its outside is rasped at this end, until it is just fully the external diameter of the platinum collar C.

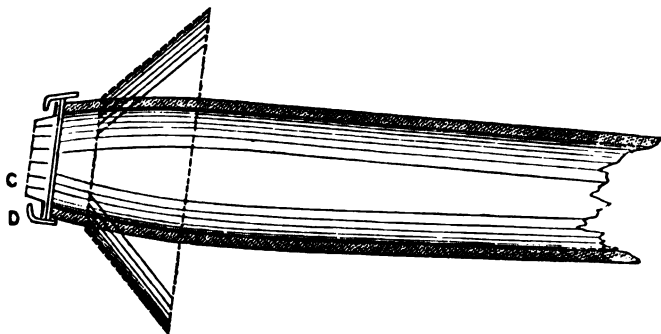
A leaden collar D, of 12 lb. lead is then to be united to the end of this pipe in the manner shown here, in section, by



the plumber's blow-pipe process. This collar D projects $\frac{3}{8}$ in. from the end of the leaden pipe. The platinum collar C, is then placed in the socket thus formed as shown in section,—



and the edge of the leaden collar D is then dressed over the edge of the platinum collar gently, and gradually all round until it clamps the face of the platinum rather than its edge closely and firmly, thus :—



This platinum collar so fixed is then ready to receive the end of the beak of the boiler, which should be inserted about 1 in. into it, and thus form a sufficiently close and durable junction between the boiler and this pipe, which slopes downwards at 1 in 12, like the platinum beak, and conveys the acid vapour and steam from the boiler to the chamber or worm or other condenser.

This leaden pipe may either form a projecting end of the worm itself or may serve as a movable adapter, by having a short elbow at its other end, dropping with a water lute on to the worm head, or it may have a short elbow looking upwards, with a water lute connecting it with a pipe leading to a chamber.

In any case, this leaden pipe, shown in the above sections, must itself be protected by water externally. This is done by laying it in a leaden trough, or in a wooden trough lined with lead, without ends, while a cone of sheet lead is united to the end where the collar D is, while its circumferential part is bent and cut square, so as to meet the edge of the leaden lining of the trough to which it is united by the blow-pipe process. The other end of the pipe and trough are connected in a somewhat similar way according to the circumstances as above stated.

Whenever the head of the boiler is to be removed, it is lifted so far upwards as to detach it from the boiler, but no higher, or the slip-joint above described would be strained; then the head is drawn back horizontally, so as to pull the beak straight out of the platinum collar C, which is in the leaden pipe. The head is put *on* by the same movements reversed. This sort of joint is preferable to a water lute in many respects.

We have now to speak of the cooler for the concentrated acid. The platinum cooler with a surrounding leaden vessel to contain water, recommended for use in connexion with this boiler, is shown in the next figure. This form of cooler for vitriol in its hottest state as it comes from the

boiler was suggested by the costliness, defects and inconveniences of the sleeved syphon or other tubes of platinum which have generally been used for this first stage of the cooling process. The advantages intended to be obtained by this cooler are less weight of platinum, greater facility for cleansing the cooler from fur externally, and from accretion and sediment internally, without the usual liability to injure the apparatus, also simplicity and compactness of construction and its consequent advantages.

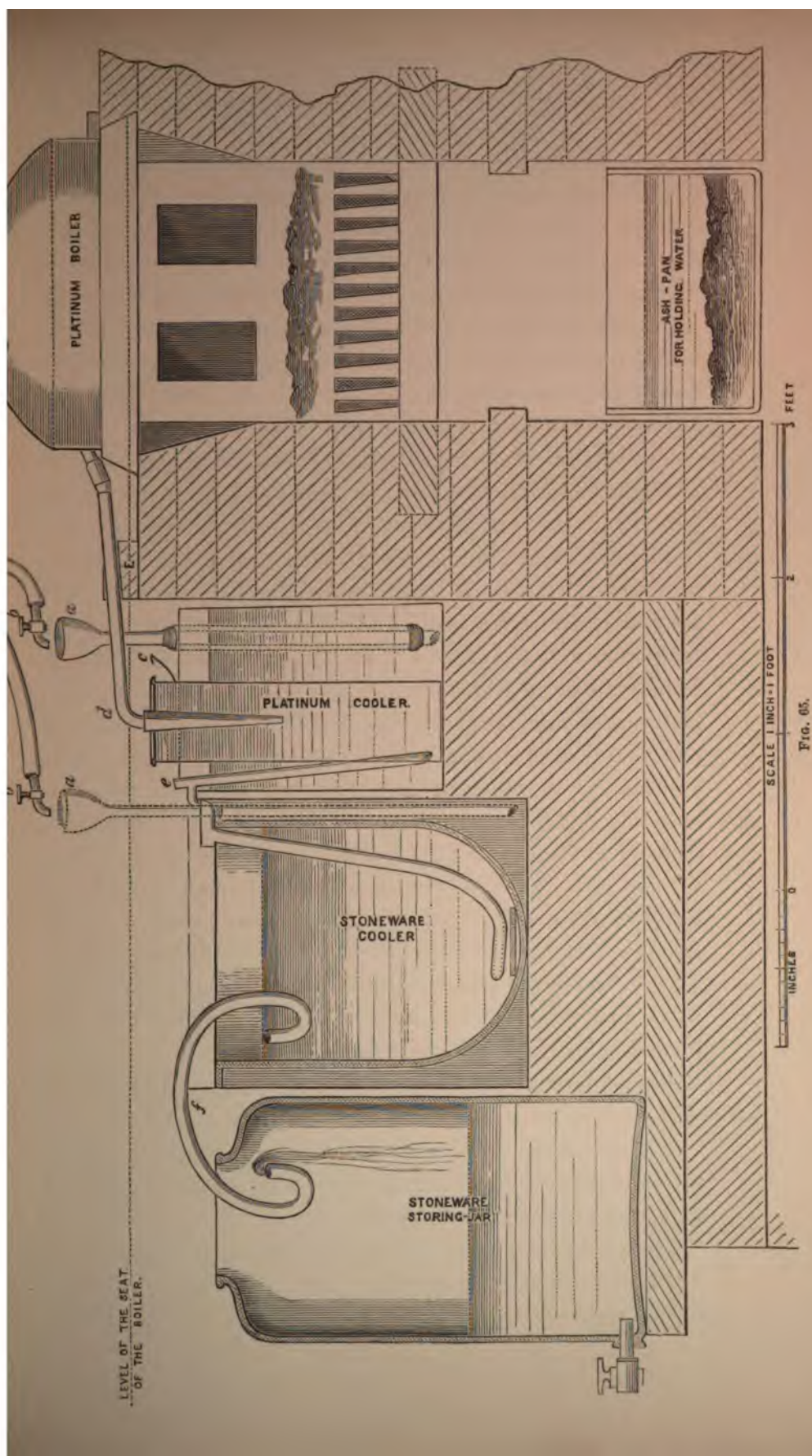
The tube which carries the tepid acid from the outflow of the platinum cooler into the next stoneware cooler or store jar, as the case may be, may be made of lead, for that metal does not materially discolour nor dissolve in acid, provided the latter be not very hot, hence for many purposes lead will serve instead of stoneware as the material for the coolers and store jar, next after the platinum cooler itself. However, the said tube, whether of lead or other material, should lead to the *bottom* of the next cooler, and rest on a slab of broken stoneware placed there, and should also be perforated at the lower end with a number of small holes, so as to emit the warm acid in a manner that shall disperse it through the mass of less warm acid in the cooler, so that it cannot rise in considerable volume, and form a warm stratum on the surface, as this would tend to crack the cooler or at least to strain it by unequal expansion. Such a leaden tube, with a receiving box at its upper end to be placed under the outflow of the platinum cooler is furnished with the other apparatus.

Fig. 65 shows a sectional view of the furnace and cooling apparatus which may be used with the platinum boiler.

a a the cold water inflow funnel made of lead. The water is led to the bottom of the leaden cooler through a leaden pipe bound round with spun-yarn or other non-conductor of heat.

b b cold water taps.

c a piece of sheet lead partially covering the leaden



cooler, so as to keep the vapour of the hot water away from the mouth or top of the platinum cooler.

d outflow pipe from the boiler. It dips 2 in. deep into the funnel of the cooler.

e outflow of cooled acid. This platinum pipe leads the acid from the bottom of the platinum cooler, and is bound round with spun-yarn to prevent the warm upper strata of water from re-heating it.

f syphon of lead or stoneware.

The outflows of water from the coolers are by lips or spouts formed in the external or leaden coolers at the proper level.

The circular leaden vessel or cylinder for retaining cold water around the stoneware cooler may be made just fully as large in diameter as will receive the top rim of the stoneware vessel. Means must of course be employed to retain the stoneware vessel down in the leaden one, to prevent its being floated up by the water, when there is not much weight of acid in it.

The syphon which discharges the acid from the stoneware cooler into the storing jar should have its ends turned up a little, so that it shall keep its charge, even if the liquor be emptied out of either or both of these vessels. It is rather advantageous also to place in the stoneware cooler, a large funnel of stoneware or lead, so long that its stem can rest on the bottom while its rim is within an inch of the top of the cooler. The leg of the syphon is dropped into this funnel, made just wide enough for the purpose, so that the vitriol is drawn into the storing jar from this, and therefore from the lowest or coolest strata of acid in the cooler.

The acid can be drawn off from the storing jar by means of the stoneware tap, into carboys, at intervals of about 2 hours, or as may be convenient.

Points to be observed when setting the Apparatus.—When the apparatus is being set in the brickwork, attention should be given to the following matters.

It is necessary to see that there be no grit or dust on the iron circle which forms the seat of the boiler; also that the said seat be smoothed by a cold chisel or old file, to remove any asperities remaining from the casting. The boiler being placed quite concentrically in the iron circle, which is first fixed quite level, take the *outflow pipe* and slip its upper orifice over the outflow nozzle of the boiler, so that this nozzle shall enter the outflow pipe for $\frac{3}{8}$ " or $\frac{3}{4}$ ", the main body of the pipe being held in its proper medium position, that is, sloping downwards at the rate of 1 in 12, and lying in a true radius from the axis of the boiler. In this position, place the end of a brick on each side of the pipe next beyond its collar, so as to prevent the pipe from sliding off the nozzle, and yet so as to allow the pipe to be raised or lowered a little at its lower end. Next place a brick (marked *E* in the last figure) under the middle part of this pipe so as to support its outflow end, which dips into the platinum cooler, at such a medium level that its outflow end can be either raised or lowered a little without causing its slip-joint to *bind* against the nozzle; this ought to be done when the slope of the pipe is about 1 in 12 as proposed.

Now place the platinum cooler (empty) in its leaden case in the manner represented in the last figure, with the platinum outflow from the cooler projecting through the space cut in the side of the leaden vessel, first taking care that the bottom of the latter be free from grit or dirt. Then move this leaden vessel containing the platinum cooler into such a position that the centre of the platinum cooler shall be just beneath the lower or outflow orifice of the outflow pipe already fixed, leading from the nozzle of the boiler. If the brickwork of the furnace prevent the approach of the leaden vessel quite so near, it may be within 2 in., and the difference can be adjusted by sliding the central platinum funnel of the cooler along its wire cradle, fixed to the top of the cooler, until this funnel comes under the lower end of the said outflow pipe.

Next, raise the leaden cooler vertically, keeping the same position horizontally, until the outflow pipe has entered the funnel of the cooler to a depth of $2\frac{1}{2}$ in. by reason of the funnel having been thus raised, while the outflow pipe remains as it was fixed. Fix the leaden cooler in this position, and level by blocks or bricks firmly placed under it. When all is thus far fixed, the end of the outflow pipe from the boiler can be raised (if required at any time) out of the funnel of the cooler, so far as to allow the funnel to be drawn aside and removed, to clear it out or examine it, without even interfering with the functions of the outflow pipe discharging its liquor into the cooler.

The lip for the overflow of the water from the leaden cooler must be made $\frac{1}{4}$ or $\frac{3}{8}$ in. below the level of the notch, through which the outflow pipe from the platinum cooler projects; so as to prevent the water from overflowing materially at this latter place.

In setting the platinum cooler in final adjustment in its leaden vessel or cooler, see that it stands quite free, without hanging by the notch through which its own outflow pipe projects, and without its pressing the latter against the leaden vessel, either sideways or forwards; then partly fill the platinum cooler with cold acid, to weight it while letting water run into the surrounding leaden cooler, and fill up the platinum cooler with the cold acid, while the water is rising in the leaden vessel.

The details of fixing the stoneware cooler and the storing jar, with the pipe, syphon, &c., belonging to them, will be sufficiently plain on reading the description of these parts in the preceding pages.

Loose pieces of sheet lead are useful to cover the surface of the leaden vessel closely around the platinum cooler contained in it to keep off the aqueous vapour, and other pieces of sheet lead to cover the top of the platinum cooler itself, to keep dust out, and to more completely hinder any acid fumes from rising, though these are usually too slight to be worth notice.

A small leaden funnel is provided also, about 3 in. in diameter, to place in the platinum funnel of the inflow of the boiler; its lower end is contracted so as just to allow the acid to pass, thus fumes are prevented from rising out of the boiler's platinum funnel, as they would otherwise do in case of the acid supplied being improperly weak.

Details in Working to be attended to.—After the whole apparatus has been fixed, as described, the following points will need attention when it is being worked.

The boiler, as has been seen, is intended to work with a continuous inflow and outflow of acid, regulated by a single tap, one from the pans, at the inflow, while the outflow is by a process of spontaneous overflow, which tends to guard against the inconveniences of the usual syphon of platinum, such as the acid being drawn off too low, or being allowed to rise too high, so that the outflow tap, in cases where it is made, does not need any attention.

The overflow nozzle before mentioned is furnished with a large tap, although it is not necessary, and such boilers have worked very well without it. This tap, therefore, is not to be regulated and watched during the working, it needs no attention as the outflow-taps of other boilers do. The use of this tap is to restrict any undue flow of acid, such as will arise from allowing relatively too little fire, or too much inflow into the boiler for a time, so as to reduce unduly the strength of the acid in the boiler, and also to stop the outflow suddenly on any emergency arising from an accident occurring to the supply of acid or to the cooling apparatus. It is therefore obvious that this tap is to be *set permanently* at such a degree of aperture as will allow a flow of acid decidedly in excess of the intended rate of working, but not inconveniently in excess; once and a half times the intended rate is generally suitable.

In setting the boiler to work, first see that there is a supply of cold water for the coolers and for the worm or other condenser of the vapour from the beak. The

quantity of water supplied will depend on the ease of obtaining it; if water be scarce, it may be supplied so scantily as to allow it to boil considerably around the worm and the platinum cooler, for the lower strata of water around the cooler will still remain cool, and will complete the cooling of the outflowing acid, provided the cooler be large in proportion to the rate of flow of the acid, while the uppermost stratum of water is being boiled away, and each ounce of steam produced absorbs five times the heat from the hot acid that an ounce of cold water would absorb in the course of being heated to the boiling-point. But where the water-supply is unrestricted, it is better to supply so much that it shall not become heated to the boiling-point before it flows away from the apparatus.

Next see that there is a supply of acid in the pans, sufficiently concentrated in the last one, while that in the others may be successively weaker. About 154° Tw. is the best strength, that being not so strong as to destroy the lead too rapidly, though about the greatest strength that can be allowed without commencing an undue rate of destruction of the lead. The advantage of not using the acid much weaker than this is that it is much cheaper to effect all the concentration possible in lead, than to do an unnecessary proportion of it in platinum.

Now charge the boiler with this acid, up to within about an inch of the outflow; next light the fire with clear burnt embers and coke until there is a good clear fire, afterwards coal may be added in front and be gradually pushed back as it becomes coked and burns clear, as has been already described. Work with a *gentle* draught to this fire, so that it shall be about an hour from lighting the fire before the acid in the boiler is concentrated. This will be indicated roughly by touching the top of the boiler with a bit of sulphur, and noticing whether the portion of melted sulphur, which will remain adhering to the boiler, turns brown and takes fire directly the bit of sulphur is withdrawn.

The point of concentration will be more accurately shown to be fully arrived at when the hand of the indicator is seen to cease to travel round, even when the indicator is gently tapped or struck with a bit of wire. When this full concentration is attained, notice accurately at what point on the dial the head end of the hand rests, make a memorandum of it, and work the boiler in future in a regular way, about 50° F. below that point, as this will be a degree of concentration which is practically considered oil of vitriol.

The precise point which the indicator shows for the utmost concentration of acid varies according to the nature of the impurities of the acid in different factories. It is generally about 680° F., after applying the proper correction for the particular indicator used. This correction is notified in writing on the underside of the dial, but its amount is of no consequence for the regular process of rectifying.

Having fully concentrated the acid in the boiler, run more in, gently at first, and gradually increase the stream by touching the tap as long as it has not the effect of bringing the hand of the indicator below or farther back than the above-named mark of 50° below the farthest indication.

When all has worked regularly for about a quarter of an hour in this way, and the rectified acid has been for some minutes running out at a small rate, then urge the fire gradually more and more, increasing the inflow of acid in like proportion. In doing this be guided by the indicator.

Do *not* increase the flow of acid into the boiler *suddenly* at any time, for, if the acid be not almost boiling, which it seldom is in the pans, a sudden increase of it in the boiler will stop the boiling, and therefore will cause the outflow to cease for a little while, and then, when it does begin to flow out again, it will flow inconveniently fast, or will rise too high in the boiler. Regularity and gradual change,

either in the intensity of the fire or in the rate of inflow, is essential in all systems of rectifying. By observing this, the rate of outflow or production may be raised to an extent dependent chiefly on the degree of attention which it is thought convenient to bestow on it.

In stopping the process at any time, it is desirable to *gradually* reduce the firing, and the rate of inflow of acid, so as to stop it entirely in the course of 20 or 30 minutes.

The "Corrugated" Form of Boilers and Pans.—A more modern form of still, securing great strength, productive power, safety and economy in working, and the highest degree of purity of acid with a minimum of platinum, is shown in fig. 66.

A A platinum pans, with corrugated bottom exposed to the flame in the flue. They can be worked in series, replacing the thick leaden pans now employed for the concentration of the chamber acid.

B platinum boiler with corrugated bottom to receive the acid at about 150° Tw. or above, from the pans *A A*, completing the concentration to 169° Tw.

C head and arm for carrying off the vapour to a leaden condenser (not shown), or directly into the chamber for utilization, if required.

D cooler to receive the concentrated oil of vitriol from the boilers and to pass it, cooled, into carboys. The double leaden water-jacket, not shown here, is so arranged that the cold water has the greatest possible amount of cooling power.

The Boiler.—By the corrugated form of bottom (Prentice's patent) the greatest possible amount of strength, surface, and consequent evaporating power, is obtained, and a considerable saving in fuel is effected.

The Pans.—By means of these vessels the large and costly leaden tanks for the previous concentration of the chamber acid, which require constant repair and renewal,



FIG. 005.

and more or less contaminate the acid, are entirely done away with. The setting of these boilers and open pans is of the simplest kind. They are placed upon an iron frame over a straight flue, and may be multiplied or enlarged to any desired capacity of production, without sacrifice of existing plant. Pans of lead (or any suitable material) of the same form or principle, which may be employed, if preferred, for the first concentration of the chamber acid, effecting a very great saving in brickwork, lead and fuel, are included in this patent.

The Cooler.—Is of an improved, economical, and convenient form, securing great cooling power with a minimum of water and space.

The total cost of such apparatus composed entirely of platinum, and therefore always of great intrinsic value in comparison with the first outlay, is less than that of any other form yet introduced.

Briefly, the chief advantages of this construction are said to be great economy in first outlay and in daily expense of working; large intrinsic value in a realizable form in proportion to the cost; purity of acid, and freedom from danger.

It will be seen that in this arrangement the acid merely flows continuously over the heated platinum surface in a shallow stream only just sufficiently deep to prevent the fire from injuring the pans and retort or boiler.

The flask form coolers are also a decided improvement, it being found that two are enough to cool the acid, so that it can be run direct into carboys. The tap between them serves to regulate the flow from the retort, and consequently the degree of concentration.



The upright shoulder of the connecting pipe between the cooling jars is widened so as to receive a small platinum spoon for taking samples of acid, to test its temperature and strength.

Setting the Apparatus.—Fig. 67 shows the setting of this arrangement of retort and pans.

- A* platinum pan.
- B* platinum boiler.
- C* boiler arm.
- D* fire door.
- E* fire bars.
- F* bearers.
- G* flue.
- H* ash pit.
- X* pan cover.

The retort is wholly of platinum. The pan itself is also of platinum, but its cover *X* may be of other material. Indeed a cover is not absolutely necessary, but it serves to economize heat.

Construction of the Pan-Cover.—Fig. 68 shows in detail the construction of a most ingenious form of cover which is found to answer admirably. It consists of a double casing of sheet copper, the outer one of which is japanned black on its exterior surface for safeguard against corrosion by acid gases. The space between the two copper casings is supplied with constant streams of cold water, entering at *a*, carried to the bottom of the cover, circulating in its interior, and making their exit as cold water at *b*. Shoulders are soldered on to the lower copper casing, to form a support for the upper one.

But for some kind of protection, this inner copper casing would be very quickly destroyed by the acid fumes inside the pan; this protection is afforded by a thin sheet of lead which withstands the acid. As the lead would soften and sag down with the heat, unless supported, it is fastened to the inner copper casing by means of little leaden buttons which are “burnt” on the lead by the autogenous process, through small holes left in the inner copper casing for that purpose. The protecting lead is turned round the edge of the copper, as shown at *c*.

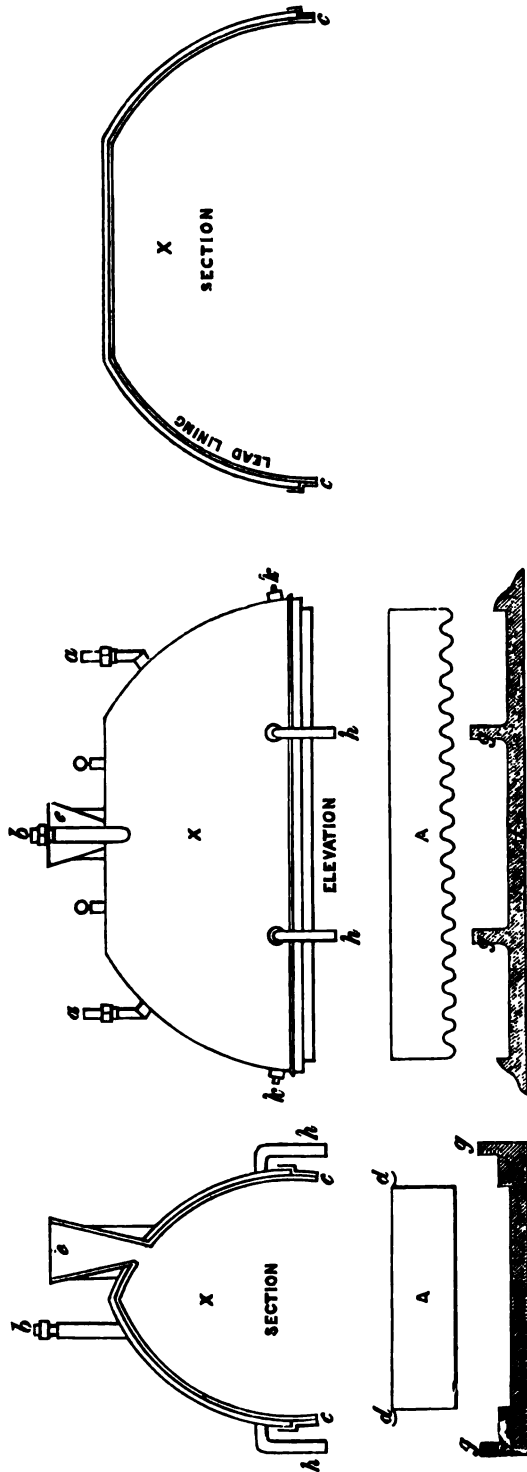


FIG. 63.

That portion of the steam arising from the ebullition of the acid in the pans *A A*, which condenses on the inside of the cover *X*, runs down into the V-shaped gutter, *d*, lutes the cover, and is drawn off by a pipe. Its strength seldom exceeds from 4°—6° Beaumé (6°—9° Tw.). The small remainder of the steam escapes by the chimney *e*; *f* are iron frames supporting the pan and retort. On the one carrying the pan *A* are cast 4 lugs, *g*, which receive *h*, the arms of the cover *X*, and thus bear the latter also; *k* are man-holes for cleaning out or seeing into the water space; *l* (fig. 67) inlet for acid; *m* outlet of concentrated acid; *n* divisions in pan bottom to make the acid circulate.

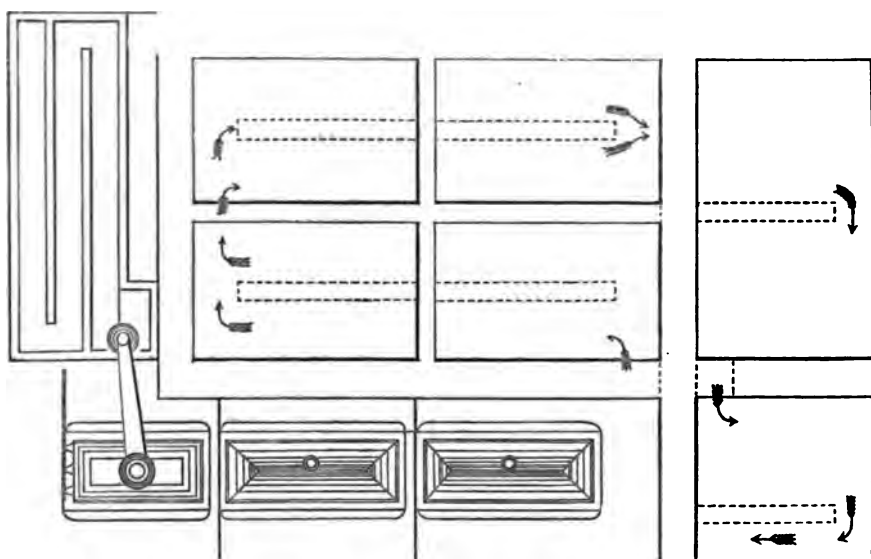


FIG. 69.

Arrangement at Griesheim.—Fig. 69 shows the arrangement adopted by Dr. Stroof at the Griesheim Chemische Fabrik, near Frankfort-on-Maine.

A to *F* are leaden concentrating pans.

G and *H* platinum concentrating pans.

K platinum boiler or retort.

The chamber acid enters *A* at 45° Beaumé (91° Tw.), and, after coursing through the 6 leaden pans, leaves *F* and enters

the first platinum pan *G* at 52° Beaumé (113° Tw.), which it leaves at 61° Beaumé (148° Tw.) running thence into *H*. It leaves *H* for the platinum boiler or retort *K* at 64° Beaumé (161° Tw.) and leaves the latter at 66° Beaumé (170° Tw.).

By employing one furnace only and leading the heat therefrom through the flues shown by the arrows underneath the whole series of the concentrating apparatus a daily make of about 6 tons of oil of vitriol, 169°—170° Tw., is obtained from this weak chamber acid (only 91° Tw.), with the consumption of less than 28 cwt. of inferior coal.

More recently, in the same works, gas has been adopted as the heating medium with very beneficial results.

We are very fortunate in being able to append Dr. Stroof's interesting report upon the working of the new system.

“Description and working results of Messrs. Johnson, Matthey, and Co.'s (London) patent corrugated bottom platinum boiler, together with a comparison of its capabilities with those of the old form.

“Messrs. Johnson, Matthey, and Co. furnished the chemical works at Griesheim, in January, June, and December, 1877, with three platinum apparatus, with corrugated bottoms, for the concentration of chamber acid (sp. gr. 1.55) to oil of vitriol, (of sp. gr. 1.843), consisting of a closed platinum boiler with platinum hood and two pans which are provided with leaden covers of my own construction. The last named are doubled lined, and allow the circulation of cold water through them in such manner that the products of the distillation are cooled and condensed. The distillate flows down the lining into the water or acid channel which is formed by the packing between the pan and the cover. From here it flows out upwards. On this lead cover is fixed a leaden tube 50 $\frac{m}{m}$ (say 2 in.) in diameter,

dipping into the water channel and rising perpendicularly out through the roof of the building. The condensed vapour which may remain in the water channel can be withdrawn therefrom. In the lead cover cold water flows in below, and is drawn off above, the cover itself is fixed in a strong iron frame, and the latter rests on four screws, so that the height can be varied with great ease.

“The platinum boiler is 900 $\frac{m}{m}$ (say 36 in.) long, and 450 $\frac{m}{m}$ broad; the two pans each 1250 $\frac{m}{m}$ (say 4 feet 2 in.) long, and 450 $\frac{m}{m}$ broad. With this apparatus is combined a leaden pan system of 5—6 pans with about 23 square metres of heat surface, under which the escaping fire gases, after having served the platinum boiler and platinum pans, are further utilized. The arrangement is so regulated that the waste heat is taken under the nearest leaden pan holding chamber acid; out of the last pan the acid flows by means of a platinum cock into the platinum pan. Here the acid reaches a temperature of 248°—266° Fahr., and has at 59° Fahr. a sp. gr. of 1.66. From the first platinum pan the acid flows at 383° Fahr., and a sp. gr. of 1.74 (at 59° Fahr.) The distillate from this pan is water, and has a temperature of 122° Fahr. A thermometer inserted through the leaden cover shows 176°—194° Fahr. Here, as also in the boiler, the acid boils up strongly. From the second pan the acid flows with a temperature from 464° to 482° Fahr., and has a sp. gr. of 1.81 at 59° Fahr. The distillate from this pan has a sp. gr. of 1.02—1.03 (4°—6° Tw.) at a temperature of 140° Fahr. The temperature under the leaden cover reaches 248°—266° Fahr., according to the supply of cold water. The acid escaping from the closed boiler through the platinum hood is completely condensed in a horizontal leaden worm, lying in water, 100 $\frac{m}{m}$ (nearly 4 in.) in diameter, and about 14 metres long. This acid shows at 59° Fahr. a sp. gr. of 1.45 (90° Tw.) and has a temperature of 140° Fahr.

"The apparatus turns out on the average 6000 kilos. (say nearly 6 tons) acid daily at 1.843 sp. gr. at 59° Fahr., and uses about 1400 kilos. ($27\frac{1}{2}$ cwt.) Westphalian coal.

"One drawback to this apparatus consisted in the fire-place, 1.80 metre long and only 500 m/m wide, necessitating the use of a long and heavy fire tool. It was thus inevitable that the tool would here and there come into contact with the corrugations of the bottom and cause scratches and bruises. Especially by the latter may the bottom easily become bulged upwards, and thus be left bare by the flowing acid to the destruction of the platinum. These disadvantages made themselves apparent now and again, and gave occasion for a modification of the system of firing. Two plans were feasible, either to place the boiler and the first pan side by side, and to furnish each with a distinct furnace, or to adopt a different mode of heating. I chose the latter method, and preferably gas heating, with which the boiler, when drawn off in December, was provided. From the experiment I am resolved subsequently to have three boilers altogether on this plan; two generators, each with 8000 square centimetres heat surface, will suffice for them, which latter are erected outside the concentration house, and thus enable the greatest cleanliness to be maintained within it. The success of the plan is beyond all question. The boiler as well as the pans, after four months' work, were laid off and examined, and the bottoms showed not the slightest crack, bruise, or buckle. The acid produced by this plan is of a constant strength, and the coal consumption is reduced to 1200 kilos. ($23\frac{1}{2}$ cwt.) of coal from the Saar basin.

"When the workmen are better accustomed to the plan, and some further changes are made in the generators, I hope to reduce the fuel still further.

"The advantages of the new system over the old are substantially as follows :—

“1. *Reduced Prime Cost.*

“A boiler of the old construction with 54 kilos. of platinum turned out in 24 hours 4200 kilos. of acid, or pro kilo. of platinum, 84 kilos. of acid.

“A boiler of the new construction with two pans and 84 kilos. platinum, 6000 kilos. of acid, or pro kilo. of platinum 158 kilos. of acid, consequently double the working capacity.

“2. *Saving on Wages.*

“Formerly one workman watched two boilers of the old construction, now two boilers of the new construction, hence a saving of wages in the proportion, as 84:120 or as 70:100. Also by the old system, one man was necessary to watch the concentration of the chamber acid in the leaden pans up to sp. gr. 1.7, which now do double the quantity, or in the proportion of 45:100.

“3. *Saving of Fuel.*

“A boiler of the old construction needed, with the concentration of the chamber acid to a sp. gr. of 1.7 in the leaden pan system, and thence in the platinum boiler, for 100 kilos. acid of sp. gr. 1.843, 38 kilos. coal.

“A boiler with two platinum pans of the new construction, from chamber acid to acid of 1.843 sp. gr., 20 kilos. coal. The economy of fuel is therefore about 48 %.

“(Signed) J. STROOF,

“*Director of the Chemical Works, Griesheim.*

“*April, 1878.*”

We have no personal experience of the coal used, but it would probably be quite safe to assume that it is not equal to Newcastle coal for heating purposes.

Delplace Retort.—The most recent of all proposed modifications in platinum retorts, so recent indeed that these pages were already passing through the press when it was brought under our notice, is due to M. Delplace.



1/8" M.M. WIDE



The apparatus is manufactured by Messrs. Johnson, Matthey, and Co., and to the unbounded courtesy of Mr. J. S. Sellon, of that firm, we are indebted for the drawings and description of it which we are enabled to lay before our readers.

Fig. 70 shows the plan, elevations, and sections of the whole apparatus, drawn to the French scale of mètres and centimètres:—1 mètre = 39.37079 in.; 1 centimètre = 0.3937079 in.

A A the first platinum boiler to receive the acid at about 60° Beaumé, and also the acid distilled over from *B B*.

B B the second platinum boiler in connexion with *A A*, in which the concentration can be completed up to a strength containing 79 per cent. of anhydride.

C C head and condensing arm to *B B*, having an outlet at *D* for strong distillate, while the weak uncondensed vapours pass forward and escape at *E*.

F F head and condensing arm to *A A*, whence the vapours escape at *G*.

H flask cooler to receive the concentrated oil of vitriol from the boiler *B B*.

a the connexion pipe between the boilers, by which the acid flows continuously from *A* to *B*.

b the connexion pipe between the second boiler *B* and the cooler *H*.

c the feed pipe of condensed distillate from the boiler *B* into the end of boiler *A*.

x the cup at the end of boiler *A*, which receives the condensed distillate from boiler *B*, as well as the acid from the pans of platinum or lead (not shown) at about 60° B.

The boiler *A* is placed at a level of 10 centimètres higher than the boiler *B*. The distillate from *B*, which should reach a strength of 62°—63° B., is condensed in the arm and recovered, being either passed into the boiler *A* for reconcentration as shown, or it may be separately utilized on account of its superior purity over the pan acid.

This arrangement was designed some years ago, and from practical experience in working it, the author has been able to introduce improvements from time to time until the present perfected form has been attained. The old leaden condensers are done away with, and their constant reparation avoided. A great advantage, too, of the rectangular form adopted for the apparatus is, that in working to the highest possible degree of concentration, no sulphate of iron can collect at the mouth of the outlet tube, as sometimes happens with other forms of boilers in use.

The great object of this form of alembic is to concentrate the acid to the highest practicable degree, and it is guaranteed, if properly mounted, to produce acid containing 79 per cent. of anhydrous acid, a degree of concentration which has never before been commercially obtained in platinum apparatus worked continuously. To make commercial oil of vitriol, generally called 66° B.,¹ but which really contains only 73 to 76 per cent. of anhydride, the distillate in this apparatus marks from 5° to 15° B., according to rate of working, the acid entering the apparatus at about 60° B. It is to be remarked that, to obtain the degree of 79 per cent. anhydride, the distillate must reach 62°—63° B.

The great value of acid of this high degree of concentration and its several economical advantages are well known. For dynamite manufacturers, as well as for numerous other industries, it is invaluable, and, without doubt, a demand for many purposes will arise from all sides as soon as such a quality can be commercially obtained. An apparatus of the older forms of the same weight as the Delplace appa-

¹ Consult the Table on p. 211. We have thought it best to leave the scale as quoted by our informant on account of the want of union among authorities as to what 66° Beaumé really is when represented as Twaddell or specific gravity.

ratus, to concentrate acid to a high degree (which does not succeed with continuous work), does not yield the fourth part in quantity of concentrated acid. In the platinum basins covered with lead, it is totally impossible, according to the most careful experiments, to reach this degree of concentration.

Hasenclever gives some particulars concerning the history of a platinum still or boiler which are not devoid of interest. The apparatus, having a capacity of 150 litres and weighing 28,548 grammes, was bought in 1865. In 1870 it was repaired, when 7891 grammes of new metal were used, but 6275 grms. of old metal were allowed for, thus increasing the weight to 30,164 grms. At the end of 1873 it weighed 28,452 grms., or a loss of 1712 grms. During the 9 years it was working it concentrated 6796 tons of sulphuric acid at 1.840. The loss therefore amounts to .252 grm. of platinum per ton of acid concentrated. The prime cost of the apparatus in Paris was 30,588.40 fr., being at the rate of 1050 fr. per kilo. The repairs effected in 1870 cost 3439.95 fr., together 34,028.35 fr. The worn-out apparatus sold at 810 fr. per kilo., yielding 23,046.12 fr. This amount, deducted from the total cost, gives a net cost of 10,982.23 fr., or, on the quantity of acid concentrated, about 1.616 fr. (say 1s. 3½d.) per 1000 kilos. acid at 1.840 sp. gr.

It is instructive to compare the above figures with the following, relating to a sulphuric acid works at Dieuze, where 2500 kilos. (say 5500 lbs.) of sulphuric acid are daily concentrated to 170° Tw. in glass retorts. The cost is estimated per 1000 kilos. (2200 lbs.) as follows:—

Coal	200 kilos. (440 lbs.)	.	.	4 marks.
Labour	„ „	.	.	3 „
Breakage	„ „	.	.	1 „
				8 marks (or say 8s.).

It is remarked that if the precaution be observed to change the glasses every 6 weeks, whether they are damaged or no, breakage may be almost entirely avoided, and the cost for glass reduced to about .75 mark (say 9*d.*)

Messrs. Faure et Kessler use a very flat platinum pan about 70 cent. diameter, over which is arranged a roomy leaden chest in which the weak acid vapours are condensed. We fail to see any advantage whatever in this apparatus over Messrs. Johnson and Matthey's system.

A series of observations upon the solubility of platinum in concentrating acid have been made by Scheurer-Kestner, who finds that platinum vessels are dissolved by acid in the following degrees. When the acid is free from nitrogen compounds and concentrated to 94 per cent., 1 grm. of platinum is dissolved by 1000 kilos. of acid; with acid 98 per cent., 7 grms.; and with 99.5 per cent., 9 grms. When the acid contains nitrous compounds, the platinum is much more strongly attacked. By alloying iridium with the platinum the action of the acid is much reduced, but the alloy is brittle and unsuited for the construction of concentrating apparatus.

In Vacuo in Leaden Vessels.—One proposition for rendering possible the higher concentration of acid in leaden pans without great damage to the lead consisted in conducting the distillation *in vacuo*, so as to reduce the temperature necessary to produce ebullition. It does not seem to have borne fruit, however. Perhaps Mons. de Hemptinne has succeeded at Molenbeeck, near Brussels, in overcoming one of the difficulties with which this plan is beset. He has constructed a vacuum pan with glass and glazed fire-clay stays which are not acted upon by the acid, and has thus enabled the leaden pan to withstand the extra atmospheric pressure while the acid (boiling-point in air 325° C.) may be boiled at from 190° to 195° C. under reduced pressure.

Forcing Air through hot Acid.—Another plan on very

different principles from the last has been suggested by J. Stoddart. The acid is heated in a leaden pan in the usual way till it reaches a temperature of 300° Fahr., when a current of atmospheric air is blown through it, the temperature being meanwhile kept up to the point mentioned by means of the fire underneath. Thus "brown acid" of 1.700 sp. gr. may be made at a temperature of 300° Fahr., and it is said that oil of vitriol of 1.850 sp. gr. may also be made in a similar manner with a temperature of 500° Fahr. or so.

This proposition has received considerable comment from F. Bode. He remarks that the boiling-point of acid at 144° Tw. is from 378° to 392° Fahr., but an acid of that strength is always obtained when run off from the pans at 365°—378° Fahr., and that according to Stoddart it would only be necessary to raise the temperature 40° C. less than is otherwise required. Also nothing is said about saving in fuel and labour. A well-adjusted pan will yield 1 cwt. of acid at 144° Tw. from 106° Tw. by the expenditure of about 16—18 lbs. of average coal, though often that quantity is used. It would require the setting forth of many conditions to enable one to say how much fuel would be required to heat acid already at 302° to 378° Fahr., but it is clear to Mr. Bode that the less expenditure of fuel would not amount to any real gain. Then, too, the air blown through must be compressed. By passing through the acid the air will become hot and thus acquire the power of taking up more vapour than when cold, but the heating of the air means the cooling of the acid, hence an increase of fuel would be required. The blowing of the air will need either fuel or manual labour. That the air may take up as much vapour as possible, it must be largely in contact with the acid, and should become as warm as can be attained. Hence a large column or deep pan of acid will be necessary. Pans generally contain only about 1 foot in depth of acid. Deeper pans have many drawbacks, such as the softness of the lead rendering

good support needful. They are best supported on cast-iron plates, though fire lumps are often used, despite their non-conductibility of heat. Pans are generally found to wear out at the bottom. To guard against spirting of the acid a cover or hood must be provided. Much acid will be carried off probably in a vesicular form by the air, and thus lost. Mr. Bode concludes (*Chemical News*, vol. xxiv. p. 84) with a series of calculations showing the loss of caloric inflicted on the acid by the heating of the air, &c.

Independent observations and experiments have been made by Mr. John Galletly, having the same end in view. He used a leaden box 18 in. long and 12 in. wide, and having a leaden shelf burnt in it the full length and nearly the whole width of the box, and at such a height that when it was just covered with a liquid the box held 5 gallons. Below this shelf a leaden pipe pierced with small holes was introduced, through which air was forced, the pipe passing along at about an inch below the surface of the acid. By this apparatus 5 gallons of acid of 1.830 sp. gr. were raised from 1.745 by a temperature of 400° Fahr. maintained for 1 hour, and by forcing 16½ cubic ft. of air through the acid. The loss of monohydrated acid was 11.9 per cent. against 8.8 per cent. in glass, which might be recovered, he says, by passing the gases or vapour through condensers or into the chambers. The air was only roughly measured, but may be taken as sufficiently accurate. The vapour generated contained at first no acid, the last ¼ was 1.070 sp. gr. The object sought was to make "oil of vitriol" in leaden vessels at the temperature ordinarily required to make "brown acid." Mr. Galletly feels satisfied that the action on leaden pans when making oil of vitriol is not due to the extra strength of the acid, but to the increased heat required to drive off the water from brown acid. He is satisfied that so much heat escapes from even the best arranged pans that there is plenty for superheating the air and propelling a small engine for forcing the air through the liquid.

The heat also would be applied directly, whereas with glass retorts it is radiated from the iron pots in which they stand. The labour of filling and emptying retorts and attending to the fires would be saved. He thinks even that oil of vitriol might almost be sold at the present price of brown acid, allowing for difference in the strength. The action on the lead by the acid was but slight, and offered no practical difficulty when the acid was derived from brimstone, though it might be when produced from pyrites.

These experiments were made some 6 or 7 years since, yet, in reply to our inquiry, Mr. Galletly writes under date Sept. 19th, 1878 :—"The process for concentrating acid has not been put into practical operation, we only make the acid required for our own use, and other questions have been engaging our attention. I am quite confident, however, from the experiments made, that the method would be found practically successful."

Steam.—Another method, which appears more promising than the last, consists in applying steam as the heating medium. It is described by Hasenclever as finding great favour among German manufacturers. The acid is poured into a wooden, lead-lined tank, 4 metres (13 ft.) long and the same width. On the bottom lie two coils of leaden pipe 45 m. (146 ft.) long, 0.03 m. ($1\frac{1}{8}$ in.) internal diameter, and .007 m. (0.273 in.) thickness of lead. Through them is forced steam under a pressure of 3 atmospheres, as long as the pans are filled with acid. In order to admit of the easy escape of the condensed water the bottom of the tank is shaped like a cut-down pyramid, the depth in the centre being .6 m. ($23\frac{1}{2}$ in.) and at the sides .3 m. ($11\frac{3}{4}$ in.). The ends of the coils, both outlet and inlet, communicate with a boiler at a lower level, fitted with a stop-cock. The inlet of the coil communicates with the boiler below the water level, the outlet with the steam chest, so that the condensed water is run back into the boiler. The operation is intermittent. The tank is filled with acid of 1.500 sp. gr., and

the steam supply maintained until it has reached 1.700 sp. gr., when it is transferred to a second similar tank. This also contains a leaden worm, through which the chamber acid is made to pass on its way to the concentration tank, thus the heat of the concentrated acid is economized.

In an apparatus of the size and dimensions alluded to it is said that 5 tons of chamber acid were concentrated to 1.700 sp. gr. in 24 hours. The steam in the boiler is at 3 atmospheres pressure, or 45 lbs. per square inch, or a temperature of 275° Fahr. The amount of fuel consumed is 9 per cent. of the acid concentrated. The boiler only requires occasional feeding. It is advisable to construct a hood at a short distance above the tank, so as to reduce the mischief which may result from the accidental bursting of one of the coils.

Advantages.—The advantages of the plan are said to be that the lower temperature prevents volatilization and loss of acid, that contamination of the acid by soot, dust, and smoke is avoided, and that the cost is reduced.

Improvements.—It is found that the pipes are attacked just where they enter the acid. Here the dust, though slight, gradually accumulates, and by capillary attraction causes the acid to rise a few centimetres above the level of the acid in the pan. This is very rapidly concentrated by the heat of the pipe, and occasions a speedy corrosion of the lead. To meet this defect a collar of lead is “burnt” on to the pipe just below the acid line, standing up all around it and opening outwards at the top. The damp dust will still accumulate on the exterior of this collar, but being of somewhat larger diameter than the pipe which it surrounds, the heat which reaches it is not sufficient to create any mischief.

Is hard or soft Lead better?—The increasing frequency of complaint concerning the want of durability of leaden pans employed in the concentration of sulphuric acid since zinc has been used in the desilverizing process, led to the

institution of some experiments anent the action of the acid at different temperatures upon samples of lead of various degrees of purity, with the result that pure and soft lead seems to be more susceptible of corrosion by hot sulphuric acid than ordinary harder and less pure metal.

Gossage's Tower.—Gossage proposed high towers filled with siliceous pebbles, down which the acid should be made to trickle while heated air was passed upwards among the flints. The Glover tower, however, somewhat similar in principle, has so many practical advantages over this plan that it is not worth detailed discussion.

Working Estimate.—The following working estimate of the cost and profit of sulphuric acid manufacture by a large firm using brimstone, and having no absorbing nor denitrating towers, will perhaps be interesting. It should be noted that the proportion of nitrate of soda used is very high, 7.8 per cent., and that the yield of sulphuric acid is equally low, being estimated at only 270 per cent.

WORKING ESTIMATE OF THE MANUFACTURE OF SULPHURIC ACID.

	COST.	£	s.	d.	£	s.	d.
80 tons of Brimstone per month @ per ton £7 .		560	0	0			
(Can be obtained direct much lower) less $2\frac{1}{2}\%$.		14	0	0			
		546	0	0			
Nitrate of Soda $6\frac{1}{4}$ tons @ £15 5s. 0d.		95	6	3	641	6	3
Wages		80	0	0			
Fuel		70	0	0			
Carriage		25	0	0			
Baskets		10	0	0			
Repairs		15	0	0			
Insurance (say)		6	0	0			
Rent (say)		10	0	0			
Sundries		30	0	0			
Wear and Tear		30	0	0	276	0	0
	Total cost	917	6	3			

YIELD.

80 tons Brimstone.—The yield per ton of
Brimstone is 2.70 tons O.V. at the lowest
computation 16.S

24.O

9.H.O

16)49(3.0625

2.70 × 80 = 216 tons O.V. of which

	£	s.	d.	£	s.	d.
Concentrated 100 tons. About 1300 Carboys						
@ per ton	5	16	8	583	6	8
Concentrated 66 tons @ 1 <i>d.</i> less 30% per lb. „	6	10	8	431	4	0
„ 50 „ @ 1 <i>d.</i> „ 25% „ „	7	0	0	350	0	0
				<u>1364</u>	<u>10</u>	<u>8</u>
6 tons Sulphate Soda @ 40 <i>s.</i>				12	0	0
Coke.				10	0	0
				<u>1386</u>	<u>10</u>	<u>8</u>
Yield per month				917	6	3
Cost per month				<u>469</u>	<u>4</u>	<u>5</u>
Profit per month					12	
				<u>5630</u>	<u>13</u>	<u>0</u>
Annual profit						

NOTE.—Some further remarks on concentration will be found at the end of Mr. Wallace's patent, relating to the manufacture of anhydrous acid, in the final chapter of this volume.

CHAPTER VI.

ECONOMY OF WASTE PRODUCTS.

Economy of Waste Products.—The waste products arising from the manufacture of sulphuric acid may nearly all be turned to some account. They consist principally of sulphate of soda from the nitre-pots, of spent pyrites, and of metals—such as lead, platinum, &c.—used in the plant of a works.

Sulphate of Soda.—The sulphate of soda residue from the nitre-pots must first be subjected to heat in a reverberatory furnace in order to drive off the sulphuric acid in excess.

The outside of the furnace is built with common bricks. The bottom of the furnace is filled up with common bricks, and grouted to within $4\frac{1}{2}$ in. of the level of the working bed. The bed and arch of the furnace are of fire-brick on edge. The insides of the fireplace and of the furnace are lined with fire-brick, $4\frac{1}{2}$ in., and tied. The upright binding rods are $3\frac{1}{2}$ in. $\times$ $2\frac{1}{2}$ in., the horizontal bars are 1 in. square wrought iron.

When the furnace is built, it should be heated strongly, but the temperature must be increased gradually during a week before using it for drying the sulphate of soda.

Let 6 or 7 cwt. be used at a charge, and keep up a good strong fire. The charge must be spread over the bed, and in about half an hour the sulphate, if containing an excess of acid, will begin to soften; then it should be turned from both ends into a heap in the middle, and thoroughly mixed, so as to have it all alike. If the sulphate is very soft when heated, a little common salt thrown in and mixed with it

will help to dry it up sooner. The charge must be fired and turned over pretty often, till the most of the free acid is driven off, which can be ascertained at first by taking out a little on a shovel, and afterwards by its appearance in the furnace. About three hours should do for a charge. Whilst working the charge, care must be taken to keep it as fine as possible by breaking the lumps whilst soft.

A considerable quantity of impure sulphuric acid gas will be driven off, and should be utilized by catching it in a condenser, or by other suitable means. It must not be allowed to go freely into the air by means of the chimney on account of creating a serious nuisance.

The dried sulphate of soda or salt cake, as it is called, is largely used for the manufacture of soda, and also in glass and ultramarine making.

Spent Pyrites.—Iron.—The burnt cinders of iron pyrites, containing no other metal of value, may be levigated, washed and dried, and the fine powder resulting be used in the preparation of a red paint of superior kind.

Copper.—Spent copper pyrites may be roasted with common salt in a reverberatory furnace having a long bed, to form chloride of copper, which is washed out of the roasted mass by the application of water and dilute hydrochloric acid, and run into tanks containing scrap iron, where the metallic copper is precipitated on heating the liquid a little.

The proportion of salt added to the spent pyrites varies from 12 to 15 per cent. The amount of unburnt sulphur remaining in the spent pyrites to be treated should be slightly in excess of the quantity of copper present; should there be a deficiency in this respect, iron pyrites may be added to supply the want.

Hargreaves' and Robinson's Patent.—Where many metals are contained in the spent ore, use may be made of Messrs. Hargreaves' and Robinson's patent, which we give in its entirety, as it also includes an improved form of kiln,

devised to utilize the greatest possible amount of sulphur from any metallic sulphide.

“The objects of this invention are,—

“First, To completely oxidize or decompose sulphides, and to utilize the greatest possible quantity of sulphur contained in metallic ores.

“And, second, To convert the oxides and sulphides of certain of the metals contained in the said ores, such as copper, zinc, tin, lead, silver, and gold into chlorides, or to render them otherwise available by the action of chlorine or hydrochloric acid gases at elevated temperatures.

“Hereafter we refer to pyrites as the metallic ore to be operated upon, but it will be obvious that the first part of our invention is equally applicable to other metallic sulphides, such as galena, blende, blue Anglesea stone, and we describe burnt or spent pyrites as the substance to be operated on under the second part of our invention.

“In carrying the first part of our invention into effect we proceed as follows:—

“(A.) We burn the pyrites in tall, vertical, or other retorts or burners, until the whole of the sulphur is oxidized. For this purpose we artificially keep up the temperature of the pyrites after the greater bulk of the sulphur is oxidized, and the heat evolved is insufficient of itself to keep up the temperature. The sulphur may be oxidized either in the form of sulphurous acid or of a sulphate of some constituent of the pyrites or other ore which is being operated upon.

“(B.) We burn or treat the pyrites in retorts or burners arranged in series or sets. The air, which may be heated, is made to pass into a retort or burner containing nearly spent pyrites kept at a temperature sufficiently high to oxidize the sulphur compounds still present. After passing through the first retort or burner the air passes through another retort or burner containing pyrites, which is not so nearly spent or burnt, and thence through any desired number of retorts or burners. Stoppers, valves and

dampers are so arranged in the passages and connexions that each retort or burner becomes in its turn the first, the intermediate, and last of the set or series. We prefer that the air should be made to enter at the retort or burner which contains the most nearly spent pyrites, and the sulphurous acid produced should flow out of the apparatus from the retort or burner which has last been charged with fresh pyrites. After the pyrites ceases to give out sulphurous acid the retort or burner is discharged if the pyrites is free from copper, zinc, tin, lead, and other metals besides iron, but if metals other than iron are contained in the pyrites, and it is desirable to obtain or utilize them, we, previously to discharging the said retort or burner, treat the spent pyrites in the method hereafter set forth in carrying the second part of our invention into effect.

“The retorts or burners which we prefer to use consist of vertical, circular, elliptical, or square tubes with bearers or false bottoms to support the pyrites, and preferably heated on the outside. The said retorts or burners may be connected together so as to form a series, or may be fed in the ordinary way, and the gas from each retort or burner be made to flow directly from each retort or burner without passing through another.

“The retorts or burners may be of iron or clay, or other suitable material.

“In carrying the second part of our said invention into effect we proceed as follows, that is to say :—

“We keep the above or other spent pyrites at a suitable temperature; or we take pyrites which has already been burnt, and heat it; the temperature which we have so far found to be suitable is one from 700° to 900° Fahrenheit. Through, over, or amongst the said spent or burnt pyrites we pass a current of chlorine or of hydrochloric acid gas or vapour, whereby any oxide of copper, zinc, lead, tin, or other metals present are converted into chlorides, or otherwise made available.

"If the pyrites or other ores contain sulphides, but in too small a quantity to afford a separate treatment for the oxidation of the said sulphide, we mix atmospheric air or oxygen with the chlorine or hydrochloric acid gas used so as to oxidize the sulphides and to form chlorides at one operation.

"The pyrites after being treated with chlorine or hydrochloric acid gas, as aforesaid, is then crushed (if not already in a sufficiently minutely divided state), and lixiviated with water to dissolve out any soluble salts, such as those of copper, zinc, and tin, and the oxide of iron remaining is then fit for use in iron manufacture and for other purposes. If salts which are insoluble in water are present, such, for instance, as sulphate of lead, we submit the ore to any suitable treatment to obtain the said lead or other metal in the form of a salt or metal.

"**Description of the Drawings.**—Fig. 71 is a vertical longitudinal section, and a horizontal section at the line A, B, of vertical burners or retorts illustrative of the first part of our invention.

"In illustration of head A, 1, 1', 1'', 1''', are retorts or burners; 2, feed doors; 3, bearers; 4, discharge door; 5, 5', 5'', 5''', outlet ways, all supposed to be open to the main gas-pipe 6; 7, fire-place; 8, uptake heating flues; 9, escape flue for heating gases. The fire-place 7 and up-take flues 8 serve to artificially maintain the temperature of the pyrites after the greater bulk of the sulphur is oxidized: 10 are teasing holes. Air, heated or otherwise, is admitted past the regulating slides 11 in the discharge doors 4.

"In illustration of head B, under which the retorts or burners are so arranged as to become the first, intermediate, and last of the series, and the evolved gases pass from one to the other, the references, given above in describing head A, apply. Four retorts or burners are here shown, that marked 1 being the first of the series. Air enters past the regulating slide 11, the discharging doors 4 and slides 11

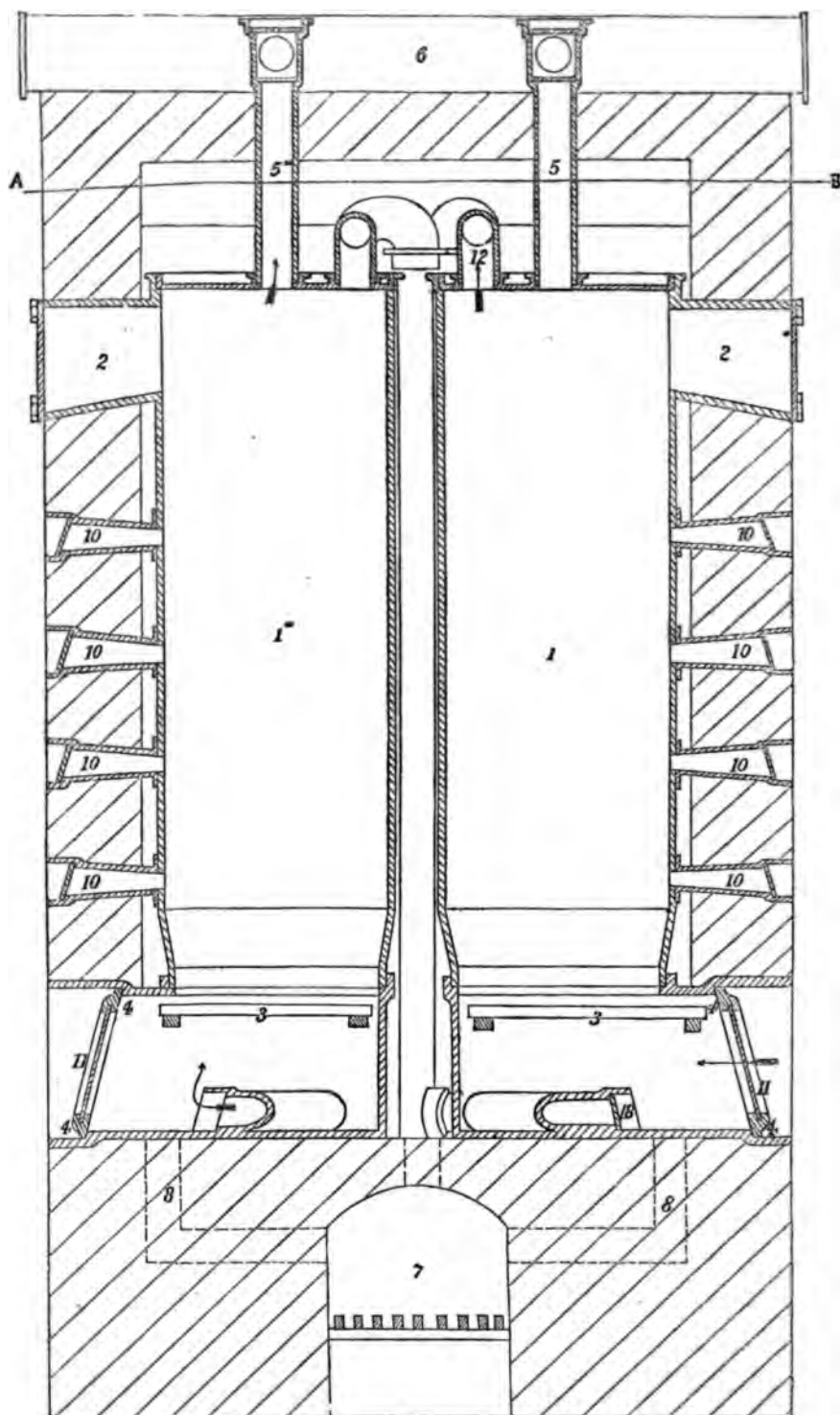


FIG. 71.

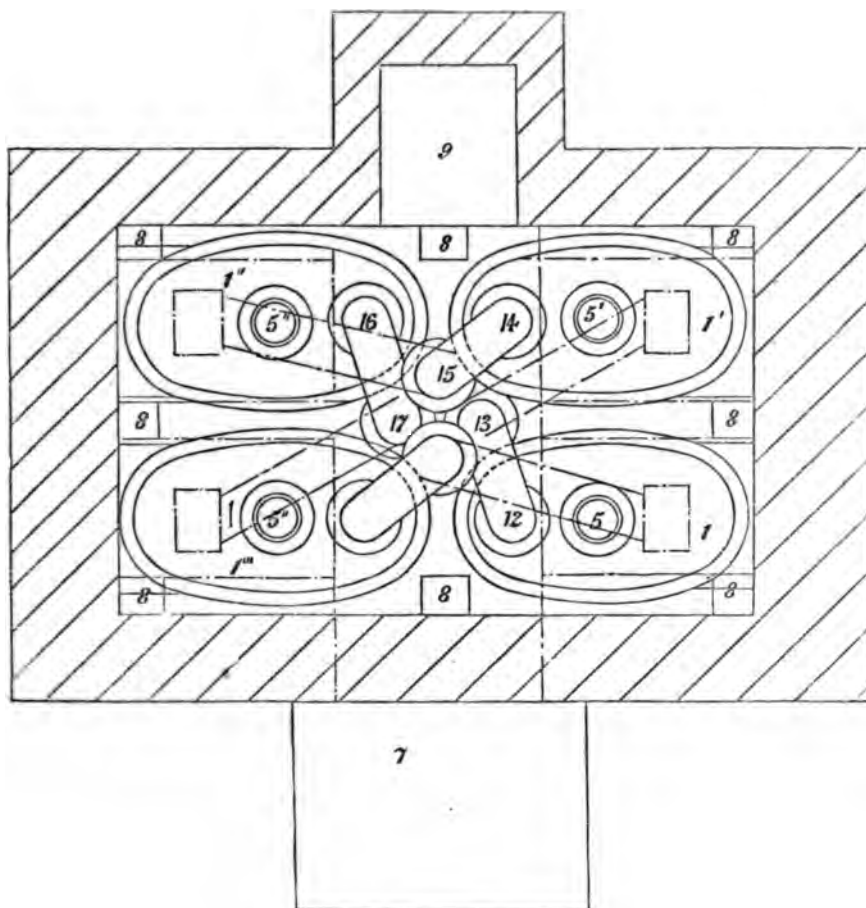


FIG. 71.

in the others being closed, and the evolved gases and air pass from 1 through the passage 12 down the pipe 13 into the bottom of retort 1', thence through the passage 14, down the pipe 15, up the retort 1'', through the passage 16, down the pipe 17, and into the retort 1''', from which latter the said gas passes through the outlet way 5''' into the main gas-pipe 6.

“It will be evident that dampers are placed in all the outlet ways except the one marked 5''', which is the last of the series. A damper is provided in the connecting passage opening into the bottom of each retort, and the one here

marked 18 at the bottom of the retort, which is the first of the series, is closed so as to prevent the air from passing direct to the outlet way 5ⁱⁱⁱ leading into the gas main 6 without passing through the whole series.

“It will be evident that by opening any particular damper in the outlet ways 5, 5ⁱ, 5ⁱⁱ, 5ⁱⁱⁱ, and closing the corresponding damper 18 in the ash-pit, that any of the chambers can be made the first, intermediate, and last of the series.

“It will also be evident that, as hereinbefore mentioned, the chamber which is the first of the series, and to which air is admitted, should be the one containing the most nearly spent pyrites, and that the one which is the last of the series should contain fresh pyrites.

“Under the second part of our invention we prefer to proceed as follows :—We admit chlorine or hydrochloric acid gas with or without atmospheric air, either after or during oxidation of the sulphur, into the retorts or chambers, and allow it to pass through the burnt pyrites contained in the series of retorts or burners. The burnt pyrites should be maintained at from 700° to 900° Fahrenheit. When the whole of the oxides of copper, zinc, lead, tin, or other metals present, iron excepted, are converted into chlorides, the mass is to be withdrawn and heated by ordinary and well-known means as herein mentioned to separate the metals. The condition of the metals, such as copper, zinc, lead, and tin, can be ascertained by withdrawing samples from time to time.”

Arsenic.—The arsenic which is present in varying degrees in almost all pyrites remains partly in the residual mass after calcination in the kiln, and is very troublesome when the cinders are to be treated as already described for the extraction of copper, for even the smallest traces of it render the copper brittle, and therefore unsaleable in the market except at a greatly reduced price.

Mr. Down in 1870 patented a plan for removing the

arsenic, but we do not know to what extent it has been availed of.

One authority says that the extraction of copper from burnt pyrites residues has checked the mining of native copper ores, and has reduced the cost of sulphur to the soda maker 40 per cent. since 1865. In 1869 the copper extracted from these residues amounted to 4100 tons on the Tyne, and about 3500 tons in Lancashire and elsewhere, and the amount of native copper ores mined in that year was only 8291 tons as against 15,968 tons in 1860.

Claudet's and Phillips's process for extracting the precious metals from burnt pyrites has been very successfully worked by them at the Widnes metal works.

Perhaps Henderson's process for copper extraction is in most general use.

Mason proposes in his patent to extract the copper first from burnt cupreous pyrites by simply well washing the residue without the application of heat, and then to subject the remaining mass to calcination, in order to drive off all residual traces of sulphur, and thus leave the iron salts in a fit state for the manufacture of metallic iron or steel.

At Meggen, in Westphalia, where the ores contain much zinc, Dr. Hofmann treats the burnt pyrites as follows. In order to get rid of the traces of sulphur which remain in the ore after burning in the ordinary kilns used in sulphuric acid works, the cinders are dried in the presence of atmospheric air, when a large proportion falls to powder, and this powder is found to be free from sulphur. The lumps, which alone contain the residual sulphur, are screened from the powder and may be reburnt. This sulphur-free powder is submitted to careful washing with water at about 104° Fahr. To these washings is then added an equivalent of common salt in excess for every equivalent of sulphuric acid present. Sulphate of soda is thus produced, and separates out as the liquid cools, and is here obtained in such quantity as to pay the total cost of the process.

After the separation and removal of the sulphate of soda, the mother liquor which remains will be found to contain chloride of zinc, chloride of sodium (common salt), and sulphates of zinc and iron. On concentrating the liquor to about 120° Tw., these various salts, except the sulphate of zinc, will be deposited. The latter can then be drawn off and used for various purposes. In Germany it is especially employed for preserving railway sleepers, and sells at about 18 fr. 75 c. per 100 kilos., or say 6s. per cwt. Metallic zinc can also be precipitated from the sulphate, if desired, by converting it first into oxide by means of lime, and then submitting the oxide to heat along with half its weight of powdered charcoal or coke dust, in a furnace properly constructed for the purpose.

The pyrites principally used in this country comes from Spain and Norway, and does not contain any zinc. Some of the Spanish ores, however, are auriferous and argentiferous as well as cupreous, while the Norwegian yield only copper and iron. The best-class Norwegian pyrites we prefer beyond all others for sulphuric-acid-making, on account of its easy management in the kilns.

Waste Metals.—Not only should all waste scraps of metal, such as platinum, copper, and lead, be carefully collected and cleaned for re-sale, but also the deposit of sulphate of lead in the chambers and other vessels may be dried carefully, to drive off the excess of sulphuric acid (which may be recovered as with the sulphate of soda), and sold to the lead-smelters. In running the scrap lead into ingots much scum will be gathered from the surface of the molten metal, which has also its value as “lead ashes.”

CHAPTER VII.

ACCESSORIES.

Thermometers.—For estimating the temperature of liquids, instruments called thermometers are used. These should be made of glass in one piece for use in acids. Sometimes vulcanite is substituted for glass in thermometer scales.

Thermometers are made on three scales, Celsius (Centigrade), Fahrenheit, and Reaumur, each in vogue in different countries. Fahrenheit is most useful as the subdivisions are more numerous.

In Celsius or Centigrade 0° represents the freezing-point of water, and 100° the boiling-point, there are therefore 100 degrees between the two points. In Fahrenheit 32° represent freezing-point, and 212° boiling-point, or 180 degrees between them. In Reaumur the two points are 0° and 80° respectively.

For converting the temperature by one scale to that of another, the following table will be found very useful :—

To convert	+	F to R	:	$R = F - 32 \times \frac{4}{9}$
„	+	F to C	:	$C = F - 32 \times \frac{5}{9}$
„	—	F to R	:	$R = F + 32 \times \frac{4}{9}$
„	—	F to C	:	$C = F + 32 \times \frac{5}{9}$
„	+	R to F	:	$F = R \times \frac{9}{4} + 32$
„	—	R to F	:	$F = R \times \frac{9}{4} - 32$
„	+	C to F	:	$F = C \times \frac{9}{5} + 32$
„	—	C to F	:	$F = C \times \frac{9}{5} - 32$
„		C to R	:	$R = C \times \frac{4}{5}$
„		R to C	:	$C = R \times \frac{5}{4}$

The sign + means above 0 (zero).

The sign — means below 0 (zero).

Hydrometers.—These are for estimating the specific gravity of any liquid, without the trouble of ascertaining

in in a scientific manner. The specific gravity of sulphuric acid increases with its strength.

They are graduated upwards or downwards from 0°, according as the liquid is lighter or heavier than water.

There are two principal scales, those of Twaddell and Beaumé. Twaddell is the better, having a greater number of divisions.

— Beaumé is graduated in an entirely arbitrary manner, and does not directly indicate the specific gravity of the liquid. The following remarks apply to the instrument for liquids heavier than water.

“ The graduation of this hydrometer is made as follows :— A solution of 10 parts of common salt in 10 parts of water is made, and the space between the point to which the instrument sinks in this liquid, and that to which it sinks in water, is divided into 10 equal parts, which rate of division is continued throughout the scale. Beaumé and others previously used 15 per cent. salt solution, and called the corresponding degree 15°, and the differences which appear in the statements of various chemists, as to the specific gravities which correspond with Beaumé’s degree, may to some extent arise from the difference in the former and present salt solutions employed in the graduation.

“ The following table shows these differences.

Beaumé.	Twaddell. 10° R.	Francœur. 10° R.	Baumen-berger. 11.5° R.	Gay-L. 10° R.	Poggiale.	Graham's chemistry.	Morveau. 10° R.
10°	1.0769	1.0704	1.069	1.075	1.075	1.073	1.075
15°	1.1200	1.1095	1.107	1.116	1.116	1.113	1.116
20°	1.1666	1.1515	1.148	1.161	1.161	1.157	1.161
30°	1.2727	1.2459	1.239	1.261	1.262	1.256	1.263
40°	1.3999	1.3571	1.347	1.384	1.383	1.375	1.384
50°	1.5555	1.4902	...	1.532	1.530	1.515	1.530
60°	1.7501	1.6522	...	1.714	1.711	1.690	1.711
66°	1.8922	1.7674	...	1.848	1.846	1.815	1.842
70°	2.0003	1.8337	...	1.946	1.946	1.909	1.942”

The appended rules for finding the specific gravity corresponding to any degree registered by Beaumé's hydrometer are due to Dr. Wilson Pile.

For liquids heavier than water :—

$$\text{Sp. gr. desired} = \frac{145}{145 - B^{\circ}}, \text{ and Beaumé desired} = 145 - \frac{145}{\text{sp. gr.}}$$

For liquids lighter than water :—

$$\text{Sp. gr. desired} = \frac{140}{B^{\circ} + 130}, \text{ and Beaumé desired} = \frac{140}{\text{sp. gr.}} - 130.$$

The conversion of Twaddell's degrees to specific gravity, and *vice versa*, is much simpler, thus :—

$$\text{Sp. gr. desired} = \text{Tw.}^{\circ} \times 5 + 1.000, \text{ as Tw. } 80^{\circ} \times 5 = .400 \\ + 1.000 = 1.400.$$

$$\text{Twaddell desired} = \text{sp. gr.} - 1.000 \div 5, \text{ as } 1.400 - 1.000 = \\ .400 \div 5 = 80^{\circ}.$$

As the temperature of the liquid also affects its specific gravity, the figures are calculated at a temperature of 60° Fahr.

For every 10° of heat by Fahrenheit above 60°, add 1° of specific gravity by Twaddell's hydrometer.

The acid to be tested should be placed in a long leaden cup, thus,—only a little more than wide enough to admit the hydrometer. Always try the temperature *before* inserting the hydrometer, as it may be high enough to break the latter instrument. Twaddell's hydrometers are divided into six grades, testing thus :—No. 1, 0°—24°; No. 2, 24°—48°; No. 3, 48°—74°; No. 4, 74°—102°; No. 5, 102°—134°; No. 6, 134°—170°. Try a low glass before using a heavy one, or it may go suddenly to the bottom, and be broken.



MANUFACTURE OF SULPHURIC ACID.

TABLE SHOWING THE SPECIFIC GRAVITY AND WEIGHT PER CUBIC FOOT OF SULPHURIC ACID BY FAUCONN'S HYDROMETER.

Specific gravity of acid at 60° F.	Weight of acid per cubic foot at 60° F.	Specific gravity of acid at 50° F.	Weight of acid per cubic foot at 50° F.	Specific gravity of acid at 40° F.	Weight of acid per cubic foot at 40° F.	Specific gravity of acid at 30° F.	Weight of acid per cubic foot at 30° F.	Specific gravity of acid at 20° F.	Weight of acid per cubic foot at 20° F.	Specific gravity of acid at 10° F.	Weight of acid per cubic foot at 10° F.	Specific gravity of acid at 0° F.	Weight of acid per cubic foot at 0° F.
1.800	115.2	1.790	115.4	1.780	115.6	1.770	115.8	1.760	116.0	1.750	116.2	1.740	116.4
1.790	115.4	1.780	115.6	1.770	115.8	1.760	116.0	1.750	116.2	1.740	116.4	1.730	116.6
1.780	115.6	1.770	115.8	1.760	116.0	1.750	116.2	1.740	116.4	1.730	116.6	1.720	116.8
1.770	115.8	1.760	116.0	1.750	116.2	1.740	116.4	1.730	116.6	1.720	116.8	1.710	117.0
1.760	116.0	1.750	116.2	1.740	116.4	1.730	116.6	1.720	116.8	1.710	117.0	1.700	117.2
1.750	116.2	1.740	116.4	1.730	116.6	1.720	116.8	1.710	117.0	1.700	117.2	1.690	117.4
1.740	116.4	1.730	116.6	1.720	116.8	1.710	117.0	1.700	117.2	1.690	117.4	1.680	117.6
1.730	116.6	1.720	116.8	1.710	117.0	1.700	117.2	1.690	117.4	1.680	117.6	1.670	117.8
1.720	116.8	1.710	117.0	1.700	117.2	1.690	117.4	1.680	117.6	1.670	117.8	1.660	118.0
1.710	117.0	1.700	117.2	1.690	117.4	1.680	117.6	1.670	117.8	1.660	118.0	1.650	118.2
1.700	117.2	1.690	117.4	1.680	117.6	1.670	117.8	1.660	118.0	1.650	118.2	1.640	118.4
1.690	117.4	1.680	117.6	1.670	117.8	1.660	118.0	1.650	118.2	1.640	118.4	1.630	118.6
1.680	117.6	1.670	117.8	1.660	118.0	1.650	118.2	1.640	118.4	1.630	118.6	1.620	118.8
1.670	117.8	1.660	118.0	1.650	118.2	1.640	118.4	1.630	118.6	1.620	118.8	1.610	119.0
1.660	118.0	1.650	118.2	1.640	118.4	1.630	118.6	1.620	118.8	1.610	119.0	1.600	119.2
1.650	118.2	1.640	118.4	1.630	118.6	1.620	118.8	1.610	119.0	1.600	119.2	1.590	119.4
1.640	118.4	1.630	118.6	1.620	118.8	1.610	119.0	1.600	119.2	1.590	119.4	1.580	119.6
1.630	118.6	1.620	118.8	1.610	119.0	1.600	119.2	1.590	119.4	1.580	119.6	1.570	119.8
1.620	118.8	1.610	119.0	1.600	119.2	1.590	119.4	1.580	119.6	1.570	119.8	1.560	120.0
1.610	119.0	1.600	119.2	1.590	119.4	1.580	119.6	1.570	119.8	1.560	120.0	1.550	120.2
1.600	119.2	1.590	119.4	1.580	119.6	1.570	119.8	1.560	120.0	1.550	120.2	1.540	120.4
1.590	119.4	1.580	119.6	1.570	119.8	1.560	120.0	1.550	120.2	1.540	120.4	1.530	120.6
1.580	119.6	1.570	119.8	1.560	120.0	1.550	120.2	1.540	120.4	1.530	120.6	1.520	120.8
1.570	119.8	1.560	120.0	1.550	120.2	1.540	120.4	1.530	120.6	1.520	120.8	1.510	121.0
1.560	120.0	1.550	120.2	1.540	120.4	1.530	120.6	1.520	120.8	1.510	121.0	1.500	121.2
1.550	120.2	1.540	120.4	1.530	120.6	1.520	120.8	1.510	121.0	1.500	121.2	1.490	121.4
1.540	120.4	1.530	120.6	1.520	120.8	1.510	121.0	1.500	121.2	1.490	121.4	1.480	121.6
1.530	120.6	1.520	120.8	1.510	121.0	1.500	121.2	1.490	121.4	1.480	121.6	1.470	121.8
1.520	120.8	1.510	121.0	1.500	121.2	1.490	121.4	1.480	121.6	1.470	121.8	1.460	122.0
1.510	121.0	1.500	121.2	1.490	121.4	1.480	121.6	1.470	121.8	1.460	122.0	1.450	122.2
1.500	121.2	1.490	121.4	1.480	121.6	1.470	121.8	1.460	122.0	1.450	122.2	1.440	122.4
1.490	121.4	1.480	121.6	1.470	121.8	1.460	122.0	1.450	122.2	1.440	122.4	1.430	122.6
1.480	121.6	1.470	121.8	1.460	122.0	1.450	122.2	1.440	122.4	1.430	122.6	1.420	122.8
1.470	121.8	1.460	122.0	1.450	122.2	1.440	122.4	1.430	122.6	1.420	122.8	1.410	123.0
1.460	122.0	1.450	122.2	1.440	122.4	1.430	122.6	1.420	122.8	1.410	123.0	1.400	123.2
1.450	122.2	1.440	122.4	1.430	122.6	1.420	122.8	1.410	123.0	1.400	123.2	1.390	123.4
1.440	122.4	1.430	122.6	1.420	122.8	1.410	123.0	1.400	123.2	1.390	123.4	1.380	123.6
1.430	122.6	1.420	122.8	1.410	123.0	1.400	123.2	1.390	123.4	1.380	123.6	1.370	123.8
1.420	122.8	1.410	123.0	1.400	123.2	1.390	123.4	1.380	123.6	1.370	123.8	1.360	124.0
1.410	123.0	1.400	123.2	1.390	123.4	1.380	123.6	1.370	123.8	1.360	124.0	1.350	124.2
1.400	123.2	1.390	123.4	1.380	123.6	1.370	123.8	1.360	124.0	1.350	124.2	1.340	124.4
1.390	123.4	1.380	123.6	1.370	123.8	1.360	124.0	1.350	124.2	1.340	124.4	1.330	124.6
1.380	123.6	1.370	123.8	1.360	124.0	1.350	124.2	1.340	124.4	1.330	124.6	1.320	124.8
1.370	123.8	1.360	124.0	1.350	124.2	1.340	124.4	1.330	124.6	1.320	124.8	1.310	125.0
1.360	124.0	1.350	124.2	1.340	124.4	1.330	124.6	1.320	124.8	1.310	125.0	1.300	125.2

The weight of sulphuric acid of various strengths per cubic foot, is found as follows, viz.: as the specific gravity of water is to the weight of water per cubic foot, so is the specific gravity of the acid (whatever it may be) to its weight per cubic foot, thus:—

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Sp. gr. water.		Weight of cubic ft. water.		Sp. gr. acid 168° Twad.		Weight of cubic ft. acid.
1.000	:	62.321 lbs.	::	1.840	:	114.67 lbs.
		1.840				
		2492840				
		498568				
		62321				
		114.670640				

DR. URE'S TABLE, SHOWING THE AMOUNT OF ANHYDROUS OR DRY ACID,
AND O.V. OR MONOHYDRATED ACID, IN SULPHURIC ACID, ACCORDING
TO ITS DENSITY OR SPECIFIC GRAVITY.

SO ₃ HO.	Specific gravity.	SO ₃ .	SO ₃ HO.	Specific gravity.	SO ₃ .	SO ₃ HO.	Specific gravity.	SO ₃ .	SO ₃ HO.	Specific gravity.	SO ₃ .
100	1.8485	81.54	75	1.6520	61.15	50	1.3884	40.77	25	1.1792	20.38
99	1.8475	80.72	74	1.6415	60.34	49	1.3788	39.95	24	1.1706	19.57
98	1.8460	79.90	73	1.6321	59.52	48	1.3697	39.14	23	1.1626	18.75
97	1.8439	79.09	72	1.6204	58.71	47	1.3612	38.32	22	1.1549	17.94
96	1.8410	78.28	71	1.6090	57.89	46	1.3530	37.51	21	1.1480	17.12
95	1.8376	77.46	70	1.5975	57.08	45	1.3440	36.69	20	1.1410	16.31
94	1.8336	76.65	69	1.5868	56.26	44	1.3345	35.83	19	1.1330	15.49
93	1.8290	75.83	68	1.5760	55.45	43	1.3255	35.06	18	1.1246	14.68
92	1.8233	75.02	67	1.5648	54.63	42	1.3165	34.25	17	1.1165	13.86
91	1.8179	74.22	66	1.5503	53.82	41	1.3080	33.43	16	1.1090	13.05
90	1.8115	73.39	65	1.5390	53.00	40	1.2999	32.61	15	1.1019	12.23
89	1.8043	72.57	64	1.5280	52.18	39	1.2913	31.80	14	1.0953	11.41
88	1.7962	71.75	63	1.5170	51.37	38	1.2826	30.98	13	1.0887	10.60
87	1.7870	70.94	62	1.5066	50.55	37	1.2740	30.17	12	1.0809	9.78
86	1.7774	70.12	61	1.4960	49.74	36	1.2654	29.35	11	1.0743	8.97
85	1.7673	69.31	60	1.4860	48.92	35	1.2572	28.54	10	1.0682	8.15
84	1.7570	68.49	59	1.4760	48.11	34	1.2490	27.72	9	1.0614	7.34
83	1.7465	67.68	58	1.4660	47.29	33	1.2409	26.91	8	1.0544	6.52
82	1.7360	66.86	57	1.4560	46.48	32	1.2334	26.09	7	1.0477	5.71
81	1.7245	66.05	56	1.4460	45.66	31	1.2260	25.28	6	1.0405	4.89
80	1.7120	65.23	55	1.4360	44.85	30	1.2184	24.45	5	1.0336	4.08
79	1.6993	64.42	54	1.4265	44.03	29	1.2108	23.65	4	1.0268	3.26
78	1.6870	63.60	53	1.4170	43.22	28	1.2032	22.83	3	1.0206	2.45
77	1.6750	62.78	52	1.4073	42.40	27	1.1956	22.01	2	1.0140	1.63
76	1.6630	61.97	51	1.3977	41.58	26	1.1876	21.20	1	1.0074	0.82

The following table of comparison between the degrees of Beaumé and Twaddell, and the corresponding specific gravities, taken from Dr. Ziureck's "Technologische Tabellen," is so much more correct than any other we have seen that it will be found of great utility.

Beaumé.	Twaddell.	Real Sp.gr.	Beaumé.	Twaddell.	Real Sp.gr.
0	0	1.000	37	69.2	1.346
1	1.4	1.007	38	71.8	1.359
2	2.8	1.014	39	74.4	1.372
3	4.4	1.022	40	76.8	1.384
4	5.8	1.029	41	79.6	1.398
5	7.2	1.036	42	82.4	1.412
6	8.8	1.044	43	85.2	1.426
7	10.4	1.052	44	88.0	1.440
8	12.0	1.060	45	90.8	1.454
9	13.4	1.067	46	94.0	1.470
10	15.0	1.075	47	97.0	1.485
11	16.6	1.083	48	100.2	1.501
12	18.2	1.091	49	103.2	1.516
13	20.0	1.100	50	106.4	1.532
14	21.2	1.106	51	109.8	1.549
15	23.2	1.116	52	113.2	1.566
16	25.0	1.125	53	116.6	1.583
17	26.8	1.134	54	120.2	1.601
18	28.6	1.143	55	123.6	1.618
19	30.4	1.152	56	127.6	1.638
20	32.4	1.162	57	131.8	1.659
21	34.2	1.171	58	135.6	1.678
22	36.0	1.180	59	139.6	1.698
23	38.0	1.190	60	143.6	1.718
24	39.8	1.199	61	147.8	1.739
25	42.0	1.210	62	152.0	1.760
26	44.2	1.221	63	156.4	1.782
27	46.2	1.231	64	160.8	1.804
28	48.4	1.242	65	165.4	1.827
29	50.4	1.252	66	170.0	1.850
30	52.2	1.261	67	174.8	1.874
31	55.0	1.275	68	179.6	1.898
32	57.2	1.286	69	184.4	1.922
33	59.6	1.298	70	189.4	1.947
34	61.3	1.309	71	194.6	1.973
35	64.2	1.321	72	200.0	2.000
36	66.8	1.334			

It is necessary to remind that the presence of foreign matters such as sulphate of lead in the acid make it apparently of higher density than the degree which corresponds to its real strength. Dr. Sprengel is said to have invented an apparatus by which the strength of chamber acid at various depths may be computed.

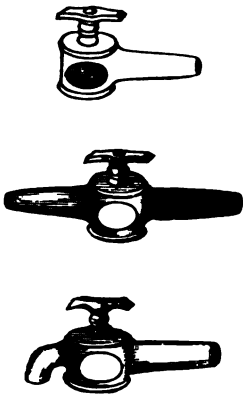


FIG. 72.



FIG. 73.

Acid Taps.—These are best made of stoneware. They are manufactured by Messrs. Doulton and Watts, Lambeth, of the shapes shown in fig. 72, and also of other patterns.

Acid Jugs.—These are also made by Messrs. Doulton and Watts, in sizes varying from 1 quart upwards as shown in Fig. 73. They are chiefly used for filling carboys from the coolers.

Carboys.—Glass carboys are generally used for conveying acid outside the works. Their size ranges from 6 to 14 gallons, but about 10 gallons is the most convenient measure, as when larger they are more liable to breakage in moving on account of their weight when filled with strong acid.

They are packed in suitable baskets made of brown rods, which should be well soaked in boiling tar and pitch, before use, or in iron baskets. Straw is used for the packing, which must be done up to the neck before filling, the remaining space being left for convenience in seeing when the carboy is full. After filling, the packing is finished by winding straw bonds round the neck and tying them down with rope-yarn. The mouths are stopped



with stoneware stoppers, which may be fastened in with a cement composed of heated sulphur and sand, or they may be capped with stiff clay and tied down firmly with sacking. The carboys should be weighed before filling, and the tare marked on each, as they vary much in weight and capacity. Be careful not to store acid in carboys where the frost can reach them, or the result will be disastrous.

Messrs. Doulton and Watts also make stoneware jars for the same purpose, which save a great deal of labour, and



FIG. 74.

are much less subject to breakage. They are becoming much used in tropical countries. Their size varies from 1 quart to 12 gallons. Fig. 74 shows their form. The stoneware stopper holds securely without any aid, it being carefully ground in, or furnished with a screw.

Steam-pipe Nozzles.—On the continent the nozzles of the pipes from which steam escapes into the chambers are made of platinum, which prevents their bore becoming enlarged, an unavoidable consequence if lead be used. The prime cost is greater, but they are very useful as permitting the inlet of steam to be regulated to a nicety.

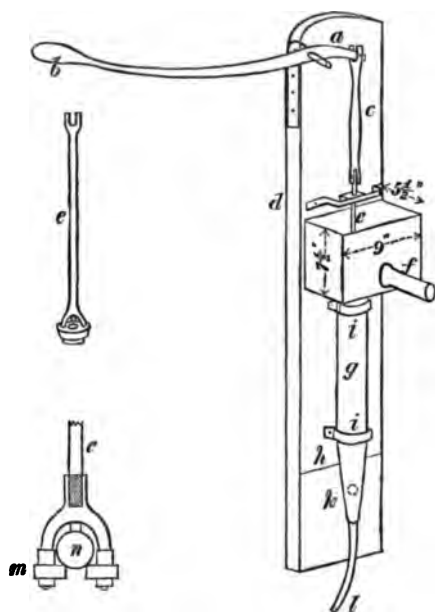


FIG. 75.

Acid Pumps.—Besides the arrangement for forcing up acid shown in fig. 49, a common leaden lift pump (fig. 75) may be used when the acid is neither strong nor hot.

Leaden Lift Pump.

a wooden plank, 5 ft. × 11 in. × 2 in.

b iron handle and support.

c iron rod.

d iron stay.

e copper plunger

rod.

f leaden box with spout $1\frac{1}{2}$ in. bore.

g leaden barrel $2\frac{1}{2}$ in. bore.

h iron plate.

- i* iron bands.
k leaden ball valve.
l leaden supply pipe $\frac{3}{4}$ in. bore.
m India-rubber packing ring.
n leaden ball valve.

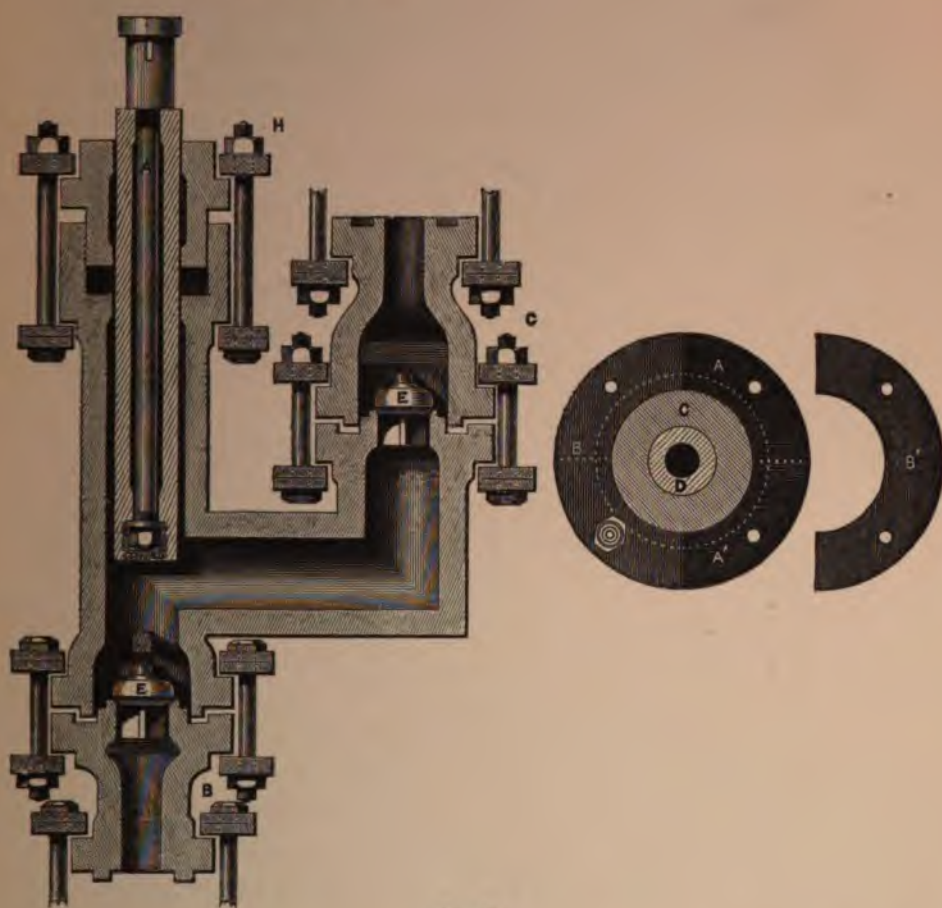


FIG. 76.

Stoneware Force-pump.—Messrs. Doulton and Watts, are the makers of stoneware force-pumps, as shown in fig. 76, which are very moderate in cost, and are admirable for forcing acid to any required height. They may be driven by hand or steam power, will not easily break, and any portion can be had separately in case of need. They are made entirely of stoneware, and packed

with asbestos. The letters on the figure are for the purpose of reference in ordering any required part of the pump.

Other Force-pumps.—Messrs. R. Daglish and Co., St. Helen's Foundry, Lancashire; and Messrs. E. R. and F. Turner, of Ipswich, sell air-compressing engines for forcing sulphuric acid without its coming into contact with the pump itself.

CHAPTER VIII.

OTHER METHODS.

Attempts to manufacture Sulphuric Acid from other Sources.—Payen gives the following notice of the various modifications that have been attempted in the manufacture of sulphuric acid :—

“ Attempts to utilize atmospheric air for the oxidation of sulphurous oxide by conducting a mixture of the two over porous substances, such as mixtures of clay with the oxides of iron and chromium, or copper and of chromium oxides, pumice, &c., have not as yet led to any useful results, and no method has yet been devised which would supersede the indirect oxidation of sulphurous oxide by nitric acid, and the use of large leaden chambers for bringing about the reaction.

“ Wöhler and Mahla suggested conducting a mixture of sulphurous oxide and air over red-hot oxides, such as a mixture of copper and chromium oxides, &c. Petrie suggested that a mixture of sulphurous oxide and air at a temperature of 300° , should be passed upwards through a high tower filled with pieces of granite kept moist with water. Persoz proposed passing sulphurous oxide into diluted nitric acid, by which the sulphurous oxide is converted into sulphuric acid and remains in solution, while the nitrogen peroxide formed escapes, and may be again converted into nitric acid by the action of air and water. In this reaction the nitric acid may be replaced by a nitrate mixed with hydrochloric acid. In the latter case, nitrogen oxydichloride is formed, which by the action of air and water is, like

nitrogen peroxide, reconverted into nitric acid. Hahner and McFarlane suggest oxidizing sulphurous oxide by chlorine in the presence of steam.

“But although such methods have not yet been rendered practicable on the large scale for the production of sulphuric acid, the oxidation of sulphurous oxide by atmospheric air, and without the intervention of nitric acid, has been so far successfully applied by Hargreaves in the direct production of sodium sulphate from sodium chloride, that his method promises to do away with the necessity for sulphuric acid in the manufacture of soda ; for which purpose greater part of the sulphuric acid now made is used. So far as this manufacture is concerned, therefore, the adoption of Hargreaves’ method would render the use of lead chambers unnecessary.

“Methods have also been proposed for separating the sulphuric acid contained in gypsum, Frémy suggesting heating gypsum with sand, and Tilghman proposing to pass steam over red-hot pieces of gypsum, the products in both cases being passed into the lead chambers. Köhsel suggests heating gypsum with charcoal, thus forming carbonic acid and calcium sulphide. The carbonic acid hereby evolved is made to decompose a solution of calcium sulphide from a former operation, the sulphuretted hydrogen thus formed being burnt, and the gaseous product conducted into the lead chambers. A number of other propositions also have been made for making use of gypsum as a source of sulphuric acid.”

Other kinds of Chambers.—Our remarks hitherto on the subject of chambers used in the manufacture of sulphuric acid have been confined to the conduct of operations in chambers formed of lead. But there are other substances which will resist the action of acids quite as well as, and indeed better than lead, such for instance as glass and stoneware, but their use has not yet become general, probably on account of their fragility, and certainly, as far as we know,

not from their unsuitability to the nature of the reactions necessary for the production of sulphuric acid. This may be especially said of glass. Every chemist knows how few substances affect good glass, and we are strongly inclined to the belief that the difference observed between the results obtained from experiments conducted in the laboratory in glass vessels and the same experiments carried out on a large scale in leaden chambers may be greatly owing to the dissimilarity of the materials forming the vessels. We know that lead is acted upon by sulphuric acid, and still more by nitrous gases, and who shall say to what extent this action upon the lead interferes with the regularity that ought to be maintained in the reactions of the gases upon each other?

Some attempts have already been made to substitute other materials for lead in the lining of the chambers, and we now propose to give details of those which appear to us to be steps in the right direction.

Ward's Plan.—The first plan we shall notice is that of Mr. Peter Ward, of Bristol, who proposed to place a condensing surface of glass inside the second of two leaden chambers. He passes the kiln gases with steam into an ordinary leaden chamber of smaller dimensions than usual (say 64 ft. $\times$ 16 ft. $\times$ 20 ft., for a consumption of 7 tons of pyrites per 24 hours), which he calls the mixing chamber, and where a certain portion of the gases will condense. The undensified gases are taken thence into a chamber 200 ft. long and 3 ft. square, in which sheets of glass are arranged horizontally and parallel to each other at a distance of about 1 in. apart, supported on strips of glass or earthenware until the pile reaches to the top of the chamber. The bottom or floor of the chamber should be cut into four equally long sections and arrangements made for running the acid out of each independently. The sheets of glass may be of any convenient size, and be placed edge to edge in piles about 4 ft. in length. After each pile an equal space is left

free for the mixture of the gases, and then another pile is made, till in this way the whole chamber is filled by alternating piles. The cover may also be made in sections for convenience in getting at the glass. Steam is introduced at the beginning of the long chamber in quantity sufficient to produce acid of 135° to 150° Twaddell, in the last section. This acid will contain a large quantity of the oxides of nitrogen, it is therefore run back into the leaden pipe which conducts the kiln gases into the first chamber, and is there made to give up the suspended nitrous gases by the combined action of the sulphurous acid and steam. Instead of sheet glass, tubes of about 1 inch in diameter may be used, piled cross-wise in the chamber. The glass should be as free as possible from alkali, or it would be slowly corroded. It may be piled as closely as the draught will admit, the closer the better. The steam must be carefully regulated: if too little, the acid will be liable in cold weather to form a crystalline mass, and if too much, the product of acid will be less and a loss of nitrous gas will also ensue.

Mr. Ward claims that by this plan a greater quantity of acid may be made in a given space, a greater product obtained from the sulphur used, a less percentage of nitre consumed, and the wear and tear of plant reduced.

We think we see one weak point in this plan, which is that though the first chamber may not suffer much wear and tear, it appears certain that the long chamber will be very rapidly corroded by the joint action of the strong nitriforous acid and the nitrous gases.

Pudney's Plan.—Eighteen months later, a Mr. Pudney of London, perhaps foreseeing this failing, patented a plan of building chambers with sheets of glass in lieu of lead. He proposed two modifications, one in which the whole structure, top, bottom and sides is lined with glass, and another wherein the bottom is of lead as in ordinary chambers. The only difficulty that exists is in joining the sheets of

glass to each other, or to the lead, as the case may be. The edges of the glass sheets are each bent downwards into leaden troughs. The troughs all communicate with each other, so that the acid, as fast as it condenses and accumulates in them, runs out by a pipe into a suitable receiver, only sufficient acid remaining in the troughs to prevent the escape of the chamber gases through the joints. The leaden troughs rest in wooden bearers, which form at the same time a framework and support the sheets of glass.

When the bottom is made of lead, a greater quantity of acid can be stored in the chamber. The sheets of glass which form the walls of the chamber are supported at their sides by uprights which are, by preference, placed at a distance of about 1 ft. 6 in. or 2 ft. apart, and are covered with lead on the parts exposed inside the chamber. The edges of this lead, after the sheets of glass are put into place, are bent round the glass, and the joint between the lead and the glass is made tight by cement or luting, and where the several sheets of glass which pass from upright to upright meet end to end, they are made to lap over each other, while the bottom corners of each sheet of glass rest on projections on the strips of lead with which the uprights are coated. The bottom edges of the lowest sheets of glass rest in troughs of sheet lead, the trough being formed by the upper edge of the leaden floor being turned up the side and bent into a U form. Condensed acid collects in it and makes a tight joint, and when it has filled the trough it overflows into the bottom of the chamber. The roof of the chamber is supported by rafters which are placed at the same distance apart as are the uprights, and whose ends rest on top plates which are carried by the uprights. The under sides of the rafters are coated with lead in a similar manner to the inner sides of the uprights and for a similar reason. They are also grooved like the uprights, to support the glass sheets forming the top. The top plates are also coated with lead on the parts

which are exposed to the acid and gases. The top sheets of glass overlap each other on meeting end to end, and thus form a tight joint, while the joints between the edges of the glass and the rafters and top plates are made tight by luting. In those cases where the joint is made tight by luting, the material which Mr. Pudney prefers to use for the purpose is composed of pipe-clay mixed with linseed oil to the consistency of glazier's putty.

Of this invention, it may be remarked that the first care would necessarily be to protect the glass from all exterior influences of weather, and every other eventuality which might injure the glass.

We believe that a combination of this plan with the one proposed by Mr. Ward, would be found to work well,—at least, that is the conclusion we have arrived at from the few practical tests we have made. Instead of a large chamber, we should suggest one shaped like Mr. Ward's second chamber, and filled with the packing of glass sheets or preferably tubes. Thus the whole apparatus, or nearly so, would be of glass.

One great gain to be derived from a chamber built of such materials would be that chamber acid might be made of a strength up to 140° or 150° Twaddell, without any extra wear and tear of plant, whereas with leaden chambers the increased destruction that would result from making acid at that strength would be disastrous. In this way those bugbears of every manufacturer—leaden concentrating pans—might be dispensed with, and instead of all the trouble entailed by them, the acid would only need denitration before being stored for alkali-making, or for concentrating in glass or platinum retorts. This denitration may either be effected in the chamber itself, or perhaps more conveniently by warming it and treating it with sulphurous acid, as described on page 136. Certain it is, at any rate, that Mr. Pudney and Mr. Ward have opened up a path which is well worthy of further exploration.

Gossage's Plans.—The next plan we shall notice is the subject of two patents taken out in 1856 and 1857, by Mr. Gossage of Widnes. Their principle seems to consist in creating a very rapid and perfect intermingling of the gases, so as to hasten the production of acid, reduce the amount of chamber space required, denitrify the acid made, and thus lessen the consumption of nitre, and at the same time concentrate the acid to a considerable degree. The latter object is brought about by a couple of towers, which differ in no material respect from the Gay-Lussac columns already described. The former end is attempted to be gained in a variety of ways.

One plan consists of a tower about 8 ft. square internal measurement, and 30 ft. high. It is simply a strongly-built leaden column lined with well-vitrified bricks laid without cement. An arch supports the packing of the tower, which consists, for a height of 10 ft. upwards, of siliceous pebbles about as large as a man's fist, and above these well-burned coke in pieces the size of pigeon's eggs is piled till within 2 ft. of the top. A leaden cistern forms an air-tight cover to the tower, and is fitted with a number of leaden pipes, provided with covers in such manner that acid can pass through them into the tower without permitting any escape of gas from it. Acid must be pumped or otherwise elevated into another cistern, and its exit thence into a "tantalus" syphon is regulated by a cock; from the "tantalus syphon" it escapes into the leaden cistern. Sulphurous acid gas from the kilns enters at the bottom of the tower, and, ascending, it comes into constant contact with the descending acid, the result being the evaporation of water from (and consequent concentration of) the acid; and as this acid is generally such as contains nitrous gases in suspension, these gases are likewise eliminated and pass with the undensified sulphurous acid gas through a pipe into the chamber, whilst a proper additional supply of nitrous acid is introduced through another pipe. The chamber is built of well-

vitrified bricks, set in a cement composed of about equal parts of sulphur and fine siliceous sand. This cement is used at such a heat as to be fluid, and the bricks should also be heated so as to ensure the strongest cohesion. The chamber is built in a dish of thick lead, which is well braced outside, and serves to collect any fluid which may escape from the chamber. The chamber is divided longitudinally into a series of smaller chambers. This division is effected by means of partitions formed of sheet glass, resting on projections formed in the side walls of the chamber, and so placed as to form inclined planes, sloping in opposite directions, alternately, and each sheet overlapping the next lower. Acid is placed in a cistern, whence it flows into a second cistern, provided with a "tantalus syphon," the exit pipe from which conducts such acid into a main pipe, from which it is conveyed, by a branch pipe, into one of the small chambers. The uncondensed gases from the chamber pass through a pipe into a tower, which is similar to the first mentioned tower, and supplied with acid from a cistern. The acid supplied must be strong (about 152° Twaddell), so as to absorb the nitrous gases passing through the tower, and this acid, thus saturated with nitrous gases, is transferred to the cistern above the chamber, and thence into the chamber as already described, and is there distributed over the inclined partitions, where it will have good opportunity of reacting upon the sulphurous acid gas, and producing sulphuric acid, and the acid thus produced is transferred to the first tower, and made to give up the suspended nitrous gases as already described.

Another plan, identical in principle, differs only in the construction of the central chamber. The chamber is heated externally in the same way, but is divided by vertical partition walls, into a series of chambers. Mr. Gossage thus describes the internal arrangements of this chamber :—

The said partition walls J and J^* are constructed of

the same materials as the external walls of the chamber *I*. The partition walls *J** have a series of openings at their lower parts communicating from the smaller chambers marked *I** to those marked *I***, and the partition walls *J* have a series of openings at their upper parts communicating from the smaller chambers marked *I*** to those marked *I**. I fill the lower part of the smaller chambers marked *I** with pieces of coke about the size of a man's fist, as high as the openings in the partition walls *J**, and upon this coke I place other pieces of coke about the size of hens' eggs, until the said smaller chambers become filled within about one foot of the top of the large chamber. In the smaller chambers marked *I*** I construct a number of arches of well-vitrified bricks set in sulphur and sand cement. These arches extend from the partition walls marked *J** to those marked *J***, and are perforated with apertures to admit the passage of gases through the same, such perforations being alternately at the sides and middle of such arches. Such perforated arches serve the purpose of repeatedly changing the currents of gases through the smaller chambers marked *I***, and thereby effecting the repeated mixing of such gases. Such mixing is also assisted by the admission of high-pressure steam through the steam jets marked *U*."

Mr. Gossage's third plan is on the same principle, and is provided with precisely the same towers, but differing in the middle chamber. This is an ordinary leaden chamber, but is fitted at its sides and ends with "blowers." They consist of tubes about 6 in. diameter, having open connexions with the chamber near the top and bottom. Into each "blower" a steam pipe with a small aperture is introduced, through which a jet of steam is passed at high pressure, by which means a considerable motion and consequent intermingling is communicated to the gases.

In the fourth and last of his plans which we shall notice, the towers differ from those previously described mainly

in these points. 1st, they are filled with very small coke, instead of having any siliceous pebbles, and 2nd, the lead casing is protected by a layer of fine coke dust instead of fire-bricks.

The connecting pipe between the tower and the chamber is of earthenware, and the supply of nitrous gas is admitted into it, instead of immediately into the chamber, thus producing a better mixture of the gases as they enter the square chamber, built of a framing of timber, and having a leaden top as an ordinary chamber. The bottom is also formed of a dish of sheet lead covered with a layer of a composition of sulphur, sand, and coke mixed, as will be hereafter described. The sides are also built of this composition in the following manner. To the framework of the chamber is attached a lining of slates covering its internal surface. A movable plank framing is then placed inside the slate lining, but so as to leave about 3 inches of space between the two linings, and into this space the melted composition is run, in sections, just as one might build a concrete wall, the same precautions being necessary with regard to ensuring the perfect cohesion of the various sections as superimposed, and care being taken to prevent the composition from sticking to the inside planking, by previously applying a thin coating of clay, mixed with water, to the surface requiring it. The inside planking is, of course, removed altogether when the walls are finished.

Pipes connecting with a steam-boiler are furnished with cocks and terminate in small orifices through which high-pressure steam is passed. Pipes, open at each end, communicate with the atmosphere and with the interior of the chamber, and are capable of being opened or closed so as to regulate the amount of air passing into the chamber. A pipe is provided for draining off acid from the chamber. The pipes are fed from the cistern with sulphuric acid (usually that containing nitrous gas), or water, which dripping through the gases in the chamber assists their admixture. The quantity of nitrous gas supplied is regu-

lated so that the uncondensed gases passing into the chamber shall contain a large proportion of free nitrous gas as indicated by their full red colour. Or, instead of thus introducing nitrous gas, liquid nitric or nitrous acid may be placed in the cistern above the chamber, with the sulphuric acid, and thence allowed to drip into the chamber, the quantity being regulated so that the gases on leaving the chamber shall have a full red colour. The acid supplied to the second tower must not be weaker than 150° Tw., and may be drawn from the first tower and then cooled. It should be in such quantity as will ensure the absorption of the whole or nearly the whole of the nitrous gas contained in the uncondensed gases passing from the chamber. Thus a large supply of nitro-sulphuric or nitri-ferous acid is obtained, which being applied as already described, "enables me to manufacture sulphuric acid with the employment of a very small proportional quantity of nitre," says Mr. Gossage.

He further claims to obtain a large supply of concentrated acid without any special expenditure for fuel, and can thus afford to apply a large quantity of such acid for the absorption of the nitrous gases so as almost entirely to prevent the escape and waste of such gases, and thereby greatly economize nitre. He reckons to supply such an amount of this concentrated acid for absorbing the nitrous gas "as shall be equivalent to not less than $\frac{3}{5}$ parts of the sulphuric acid so being manufactured." The dimensions of the towers should be such that the number which expresses in square feet their horizontal sectional area be not less than one three-hundredth part of the number which equals the capacity of the chamber expressed in cubic feet.

In preparing the above-mentioned composition a portion of good Sicilian brimstone is melted in an iron vessel and about an equal weight of pure siliceous sand is added; then for every 1 cwt. of brimstone is added about three

quarters of a cubic foot of hard, well-burned coke, previously crushed, passed through a half-inch riddle, and freed of dust by a $\frac{1}{8}$ in. sieve. Heat is then applied, and the mixture well stirred till about 340° Fahr. is reached. A sample is then run into a slab for testing. If the slab, on breaking, appear porous, more sulphur and sand must be added; if closer than necessary, more coke may be used as long as the composition be free from porosity. It is applied as explained, at a temperature of about 300° to 340° Fahr. "Apparatus constructed with an interior coating or plaster of such composition has a great capability of resisting the destructive action of acid liquids and gases; consequently provides the means of increasing the production of sulphuric acid by using much nitrous gas in its manufacture without occasioning destruction of such apparatus."

Simon has patented the following composition to be used in similar constructions. He prefers the waste stoneware of broken demijohns which have been manufactured to contain acid, and broken glass. This he mixes with sulphur in the proportions of 19 lbs. of the latter to 42 lbs. of the former. The sulphur is melted before mixing, the compound is made homogeneous and then run into blocks.

Caoutchouc has also been proposed as a substitute for lead, but does not seem to promise great results.

French Plan.—A plan of French origin was patented in 1863 by Mr. Clark, a patent agent, the principle of which is the application of abundant condensing surface—other than lead—to the action of the chamber gases; and producing concentrated acid in the substitute for chambers. Mr. Clark's description of his apparatus, &c., is so concise that we prefer to give it in his own words:—

"The manufacture of sulphuric acid has hitherto necessitated the employment of leaden chambers, the construction of which is enormously expensive, and can only be met by large capital; it is for this reason that the manu-

facture on a small scale has hitherto failed. Besides the expense of the materials themselves, the construction of these chambers requires a large plant and buildings of considerable area.

“According to this invention I dispense entirely with the use of leaden vessels in the manufacture of sulphuric acid, which may thus be readily made even by those who are not manufacturers, but still use large quantities of this acid, and thus obtain it at less cost. Although I dispense with lead chambers, the improved apparatus of this invention nevertheless presents a large surface to the gas, and so permits of its action on one and the other with the same facility as with lead chambers.

“These improvements are based on the principle that the combination of gases or vapours may be easily effected under the influence and through the intermediation of porous bodies. The substance I employ for multiplying the points of contact of the gas, and presenting a large surface by its porosity in a small volume, consists of coke which is well washed and calcined, the apparatus being formed of earthenware and of peculiar form. The production of the sulphuric acid is effected in the ordinary manner, that is to say, by the combustion either of sulphur or of iron or copper pyrites. This combustion is effected in furnaces which are not shown in the annexed drawing (fig. 77), and, as well known in such manufacture, the azotate of soda is decomposed in said furnaces by means of sulphuric acid in order to produce azotic acid, necessary for the transformation of sulphurous acid into sulphuric acid. On leaving the furnaces the sulphurous acid vapour and the azotic acid pass through sheet-iron pipes into a central brick chimney and thence through a large cast-iron pipe into a leaden pipe dipping into a bath filled with water for cooling the gas. On their exit from the tube the gases pass through the long bent pipe *G G*, having at its lower part *H* a cock, *O*, for drawing off

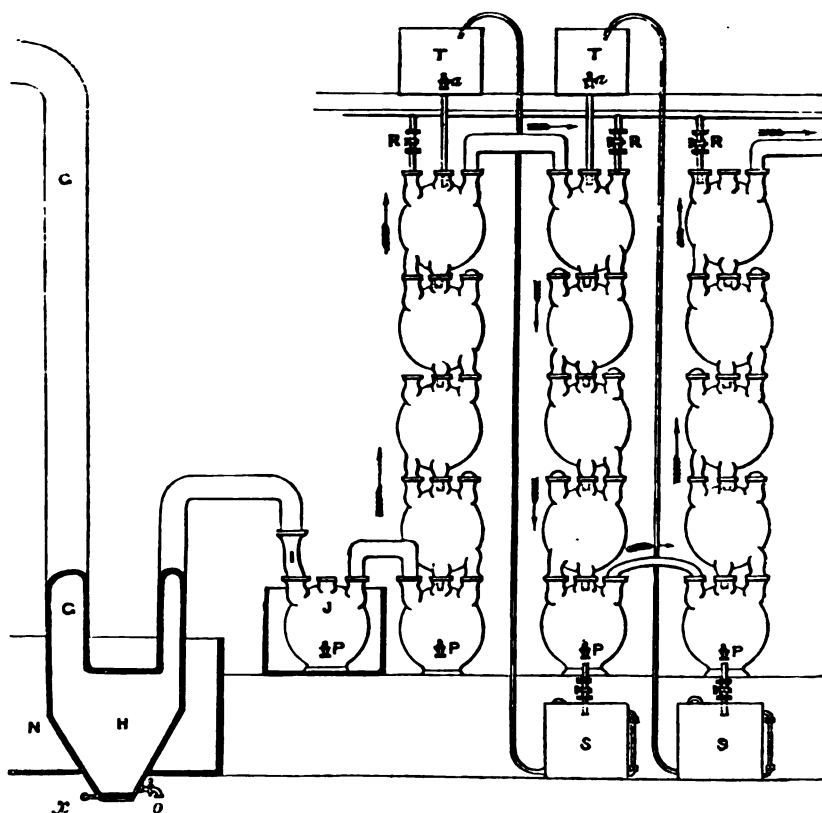


FIG. 77.

the condensed liquids. The part *H*, which is much larger than *G*, dips in a bath, *N*, filled with water, and also intended to cool the gas and retain any dust and arsenious and selenious acids ordinarily contained in pyrites. In order to prevent the dust and acids produced by the combustion of the pyrites from being drawn into the other parts of the apparatus, I apply at bottom of tube *G* in part *H* a small leaden trap, *X*, which projects below the bottom of bath *N*, and by which all the impurities may be easily removed as they are deposited thereon; on leaving part *H* the gases pass into a stone pipe, *I*, in communication with a chamber *J*, three-fourths filled with coke. The vapours thence pass in the direction of the arrows through four or five series of chambers all filled with coke.

The lower series of chambers are each furnished with cocks, *P P*, through which the condensed sulphuric acid passes into the leaden baths *S S*. In order to give greater density to the acid in baths *S*, I raise it by means of air or steam pressure into baths *T T*, through which, by means of cocks *a*, the acid produced in the last series of apparatus passes into another series, nearer the furnaces, and so on until it arrives at the first series, where it acquires a high degree of concentration, and in order to facilitate the action of the gas I also inject a jet of steam into the chambers by means of cocks *R R*. On leaving the last row of chambers the gases, which are entirely deprived of sulphurous and nitrous acids, and containing none but inert gases, are passed out at the chimney."

In alluding to the foregoing several novel plans for supplanting leaden chambers, we do not wish to be misunderstood. On a previous page we have recorded our experience that in working with an ordinary *leaden* chamber, we found that *space* was more necessary than *surface*, and universal practical opinion corroborates us. In the class of chambers (or substitutes for chambers) we have just described, however, we find that *space* is not considered so beneficial as *surface*. Here is an apparent contradiction of principle. We say apparent, for does it not rather seem that what the gases really require is *an opportunity of coming constantly and repeatedly into contact with each other*? This opportunity is afforded them, by means of space and time and jets of steam at high pressure in leaden chambers, and by means of constant concussion with obstacles in the case of small towers, trunks, boxes, &c., packed with obstructive materials.

As we have already hinted, the different natures of the surfaces on which the gases impinge may also be an important factor in the sum.

It is far from our purpose to advocate theory *versus* practice, and we should strongly dissuade any one just com-

mencing a sulphuric acid works, *with the object of making it a commercial success*, from attempting to attain his object by adopting off-hand the newest inventions or suggestions, for he would probably become much poorer, and but little the wiser for his experiment ; but some of our leading manufacturers, whom the possible risk of losing a few thousand pounds in research would scarcely affect, might, in our opinion, advance the improvement of the manufacture many stages by carefully prosecuting the subject on the lines laid down by Messrs. Ward, Pudney, Gossage, and Clark.

CHAPTER IX.

NORDHAUSEN OR FUMING ACID AND SULPHURIC ANHYDRIDE.

A VARIETY of sulphuric acid differing in some essential points from the ordinary, or as it is sometimes called, English acid, is that known either as fuming acid, from its property of giving off white fumes when exposed to the air, or as Nordhausen acid, from the small Saxon town where the industry was developed at a very early date, long prior, in fact, to the adoption of the present general mode of manufacture.

The present seat of the manufacture of this acid is at Pilsen and Elbogen in Bohemia, where one individual appears to enjoy a monopoly, possessing 12 establishments with 110 furnaces, working 72,400 retorts, and yielding annually about 34,000 quintals (say about 3350 tons) of the acid. It is an oily liquid composed of a mixture of variable quantities of sulphuric anhydride and monohydrated sulphuric acid, or anhydrous acid dissolved in ordinary sulphuric acid.

The most common mode hitherto followed in the manufacture of this acid is dependent upon the fact that basic and neutral sulphates of iron when heated to redness decompose principally into oxide of iron, and sulphuric anhydride. The heat applied is at first gentle, in order to drive off the water, and afterwards it is raised and maintained so as to distil the anhydrous fumes, which are caught in suitable receivers furnished with a little ordinary sulphuric acid which absorbs the anhydride. The oxide of iron residue remaining in the retorts is treated with ordinary sulphuric

acid, to produce a fresh supply of ferric sulphate. Such is the method chiefly pursued in Bohemia.

In France sodium bisulphate is gradually coming into favour as a source of the acid, the operation and apparatus being analogous to those current in Bohemia.

Until very recently the principal and almost sole use of fuming acid was for dissolving indigo, which it is capable of doing without destroying the colour, but it is now in fast increasing demand for employment in the manufacture of aniline dyes, and of ozokerite, and competition is being excited in its production.

Winckler adopts another principle in the manufacture, viz. resolving concentrated sulphuric acid into sulphurous acid, oxygen, and water by the application of great heat. The water is condensed, and the two gaseous products remaining are brought into contact in the presence of a porous body heated to redness: he uses platinized asbestos when they recombine to form sulphuric anhydride. The platinized asbestos is said only to require heating at the commencement of the operation, as the heat generated by the combination of the gases maintains it afterwards at a sufficiently high temperature. A furnace with 5 retorts treats 1800 kilos. (about $35\frac{1}{2}$ cwt.) of concentrated sulphuric acid daily, yielding 1000 kilos. (say 1 ton) of sulphuric anhydride, which, if dissolved in 1500 kilos. of ordinary sulphuric acid 170° Tw., will produce 2500 kilos. of fuming acid containing 85 per cent. of SO^2 .

As we have seen, one step in the process consists in the formation of sulphuric anhydride or anhydrous sulphuric acid, i. e. acid absolutely free from water, whereas ordinary concentrated sulphuric acid of 170° Tw. contains still about $18\frac{1}{2}$ per cent. of water. Now it is quite feasible to procure this anhydrous acid in a pure state instead of dissolving it in ordinary sulphuric acid to form fuming acid. It is only necessary to catch the fumes in a dry receiver placed in ice. It then forms white crystals much resembling

asbestos. These melt at 66° Fahr., and boil at 120° Fahr. They exhibit no acid properties unless moisture be present. Combination with water is so violent as to produce explosion.

It has lately been sent over to this country from Germany packed in tin cases, which it does not affect under ordinary proper precautions. It is said to be sold at Bonn on the Rhine at 8 thalers per kilo. net, or nearly 11s. per lb.

Already several patents have been applied for or granted concerning the manufacture in this country.

Squire proposes to distil the highest concentrated sulphuric acid, and to pass its vapour over a highly heated surface, such as a platinum tube raised to bright red or even white heat. The tube may be empty or partially full of platinum or pumice, &c. Platinum tubes are best as requiring less heat, but stoneware may also be used. The sulphuric acid vapour is thus decomposed into sulphurous acid, oxygen, and water. These are passed through a leaden worm lying in cold water, which causes a condensation of the water or most of it. The remaining two gases, oxygen and sulphurous oxide, are then carried over or through sulphuric acid in order to dry them. This may be conveniently effected in a coke tower saturated with sulphuric acid such as the Gay-Lussac absorber. The dry mixture of the gases is then made to combine by the action of platinum sponge or black, pumice, asbestos, certain metallic oxides and compounds, as the oxides and subsulphates of iron, chromium, copper, &c., either alone or in combination, exposed to dull red heat arranged in tubes or in a chamber of platinum or stoneware, called a "recomposer." Sulphurous oxide (SO_2) and oxygen (O) produce sulphuric anhydride (SO_3). This may be condensed by itself in a suitable apparatus, or be absorbed by ordinary acid, to produce fuming acid as already indicated. Whatever escapes of sulphurous oxide and oxygen in an uncondensed form is passed through a second recomposer, and thus saved.

Another method is suggested by Dr. Messel. Sulphur is heated in the presence of oxygen so as to produce sulphurous acid, and the gases are then passed into a suitable gas-holder, into which is admitted such an excess of oxygen as will, with the generated sulphurous oxide, form an equivalent proportion for their conversion into sulphuric anhydride, and should these equivalent proportions not be immediately attained, such proportion of either gas as may be necessary to complete the perfection of the combination may be subsequently introduced. The mixed gases are then treated substantially as indicated by Squire.

The oxygen required is to be produced by the decomposition of acidulated water by electricity derived from an electro-dynamic machine. The hydrogen from the decomposed water may be used for heating purposes or, after being carburetted, for lighting. The process may be rendered continuous by having 2 gas-holders.

The subject of a third Specification (Sonstadt) is the substitution of magnesium sulphate (Epsom salt) for ferric sulphate. The salt is heated to redness to drive off the water of crystallization, and the anhydrous sulphate is then treated as ferric sulphate for producing sulphuric anhydride. It is claimed that the magnesium salt process is cheaper, and the yield of anhydride greater in accordance with the difference in the weights of the combining substances.

Decidedly practical ideas are contained in Mr. Wallace's (of Battersea) patent.

The bisulphate of soda is placed in large retorts of glazed plumbago, preparation of earthenware, or any substance not liable to be decomposed, nor to decompose the anhydride itself. The retorts are first heated to dull redness, when any free sulphuric acid and water are driven off. The water is condensed in a leaden worm, and when the anhydride commences to distil over, the passage of the gases into the leaden coil is stopped by a three-way cock, and they are passed into a series of receivers or Woulfe's

bottles, constructed so as to retain no solid sulphuric anhydride in the lutes. The tops of these receivers are made detachable, so that the product can be removed in them (with proper covers), and thus may be avoided the ill effect upon the workmen engaged in transferring the acid from one vessel to another, occasioned by the contact of air with the acid crystals.

The process is made continuous by introducing the requisite quantity of warm sulphuric acid into the retort before the residue of neutral sulphate of soda has become solidified. Thus the bisulphate is formed, and once produced therein, it need never be removed.

An absolutely anhydrous solid acid is obtained without any of the drawbacks attending the manufacture as carried on in Germany.

Other bisulphates may also be used, but they are inferior.

Again he proposes the distillation of sulphuric acid of 1.850 sp. gr., in stills of glazed plumbago, passing the gas through tubes of the same material heated to redness. The sulphuric acid or water remaining (if any) may be absorbed in scrubbers of chloride of lime, or of pumice moistened with sulphuric acid.

Further, sulphur may be burnt in oxygen in iron or other pans, the oxygen being regulated by a weighted valve so as to get a continuous pressure. Direct combination of the sulphurous acid and oxygen is also obtained by passing the gases through glazed plumbago tubes filled with platinized pumice, asbestos, or spongy platinum kept at a red heat. Plumbago glazed inside is perhaps the best substance, for the platinum is objectionable on account of the thickness required consuming a considerable weight of that expensive metal. The anhydride is condensed in receivers as before.

The same specification contains an account of a novel form of apparatus for concentrating ordinary sulphuric acid. A coil of iron pipes are supplied with air at 700° Fahr., which

is distributed by means of platinum or porcelain pipes into a still formed of glass, glazed plumbago, platinum, &c. The still may rest on the iron coil, while the inner coil is perforated so that the acid within the still is subjected to a double amount of heat. The still also may be provided with a false bottom. The hot air is drawn through the acid by means of an aspirator or an air force-pump. The weak acid fumes driven off are condensed, and pumped up for injection into the chambers as spray or atomized acid, thus saving fuel for generating steam. Sulphuric acid is made to replace water in the cooler.

NOTE.—We believe we were with the majority in supposing that Dr. Messel's discoveries were subsequent to Winckler's. The former gentleman has, however, been good enough to give us an opportunity of satisfying ourselves that he was at least contemporaneous with, if not prior to, Winckler in his investigations.

He also claims equal credit with Squire, whose collaborateur he formerly was, for the process ascribed to him.

The method of manufacturing anhydride by the decomposition of ordinary concentrated sulphuric acid has been found by Dr. Messel to be the cheapest and best, and is the only one pursued by his firm on a commercial scale.

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LONDON:
GILBERT AND RIVINGTON, PRINTERS,
ST. JOHN'S SQUARE.

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